EXHIBIT D

ANALYTICAL METHODS FOR AROCLORS

Exhibit D - Analytical Methods for Aroclors
Table of Contents

1.0 SCOPE AND APPLICATION

1.1 The analytical method that follows is designed to analyze water, soil, sediment, other solid matrices, oil and oily sludge samples from hazardous waste sites for the Aroclors on the Target Compound List (Exhibit C, PCB). The method can be used for determining analyte concentrations in the range from the contract required quantitation limits (CRQL) to one million times the CRQL in these matrices when appropriate dilutions are made. The method is based on the Contract Laboratory Program Statement of Work, OLM03.1, and it covers sample extraction, extract cleanup techniques and Gas Chromatography/Electron Capture Detector (GC/ECD) analysis for Aroclors.

1.1.2 The target compound list may be designated as all Aroclors listed in Exhibit C, PCB, or a subset of that list and will be indicated on the chain of custody form accompanying each sample delivery group (SDG). If the site specific Aroclor(s) are known at the onset of the sampling event, the site specific Aroclor will be specified to the Contractor either by the RSCC or on the chain of custody form accompanying each sample delivery group.

2.0 SUMMARY OF METHOD

2.1 Water Samples

Continuous liquid-liquid or separatory funnel extraction procedures are employed for aqueous samples. A 1 L volume of sample is spiked with the surrogate solution and extracted with methylene chloride using a separatory funnel or a continuous liquid-liquid extractor. The methylene chloride extract is dried with anhydrous sodium sulfate, solvent-exchanged into hexane, and concentrated to 10 ml final volume. A 1 ml portion of the extract is subjected to a sulfuric acid cleanup and the resulting extract is analyzed using a dual capillary column Gas Chromatography/ Electron Capture Detector (GC/ECD) technique.

2.2 Soil/Sediment/Solid Samples

A 30 g aliquot of sample is spiked with the surrogate solution, mixed with anhydrous sodium sulfate and extracted with a 1:1 acetone/methylene chloride solvent mixture by sonication. The extract is filtered, concentrated, solvent-exchanged into hexane and brought to final volume of 10 ml. A 1 ml portion of the extract is subjected to a sulfuric acid cleanup and the resulting extract is analyzed using a dual capillary column capillary Gas Chromatography/Electron Capture Detector (GC/ECD) technique.

2.3 Oil and Oily Sludge (Waste)

A 1 g portion of sample is spiked with surrogate solution and quantitatively diluted with hexane to a final volume of 10 ml. A 1 ml portion of the extract is subjected to a sulfuric acid cleanup and the resulting extract is analyzed using a dual capillary column capillary Gas Chromatography/Electron Capture Detector (GC/ECD) technique.

2.4 Method Detection Limits

Prior to analysis, method detection limits (MDLs) for the combined Aroclors 1016/1260, must be established in accordance with 40 Code of Federal Regulations, Part 136, Appendix B. Separate MDL studies may be required by the Agency for site specific Aroclors. All MDL values must be less than or equal to one-third of the CRQL. The MDL study must be conducted using the same specifications as for sample analysis. These specifications include but are not limited to: extraction method, initial and continuing calibration conditions and technical acceptance criteria and all instrument operating conditions. The MDL study must be conducted prior to sample analysis, for each alternate column/technique and/or at least annually, whichever, is more frequent. Seven aliquots of reagent water and/or appropriate clean matrix (such as muffled sand) spiked at 3-5 times the expected MDL are analyzed. Separate MDL studies must be conducted for aqueous (either continuous liquid-liquid or separatory funnel extraction) and soil/sediment/solid sonication methods. An MDL study is not required for waste dilutions. The MDL for Aroclor 1016 will be applied to Aroclors 1221, 1232 and 1242. The MDL for Aroclor 1260 will be applied to Aroclors 1248, 1254, 1262 and 1268. All sequential analyses of MDL standards must be reported and used in the resulting MDL values which are calculated. The MDL results are calculated as described in 40 CFR, Part 136, Appendix B and reported as a separate SDG in accordance with Exhibit B. The appropriate Students' t value must be clearly provided with the algorithm used to calculate the MDL values. MDLs shall be determined and reported for each instrument/column and method. In addition to these requirements, the Contractor may provide copies of previous MDL studies for other Aroclors, which have been completed within the past six months of this request.

The MDL study must be reported as detailed in Exhibit B. The individual analytical sequence raw data must be provided and these data must be summarized in a table which demonstrates the calculated MDL values. The summarized MDL results table must include the concentration found for each compound in each aliquot, the mean concentration of each compound, the percent recovery of each compound, the standard deviation for each compound, and the Method Detection Limit. The true concentration of the compound in the spike solution must also be provided. The table must list the compounds in the same order as they appear in the target

compound list in Exhibit C. In addition, the MDL values for each instrument and method used in reporting results for an SDG shall be submitted with each data package.

The annually determined MDL for an instrument and method shall always be used as the MDL for that instrument/method during that year. If the instrument/method is adjusted in any way that may affect the MDL, the MDL for that instrument/method must be redetermined and the results submitted for use as the established MDL for that instrument/method for the remainder of the year.

3.0 DEFINITIONS

See Exhibit G for a complete list of definitions.

4.0 INTERFERENCES

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. These contaminants lead to discrete artifacts or to elevated baselines in gas chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the extraction and analysis conditions of the method by running laboratory blanks as defined in Section 12.0. Interferences caused by phthalate esters can pose a major problem in analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the site being sampled. The cleanup procedures must be used to remove such interferences in order to achieve the contract required quantitation limits.

5.0 SAFETY

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical should be treated as a potential health hazard. Exposure to these reagents should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be made available to all personnel involved in these analyses. Specifically, concentrated sulfuric acid and the 10 N sodium hydroxide solution are moderately toxic and extremely irritating to skin and mucous membranes. Use these reagents in a fume hood whenever possible and if eye or skin contact occurs, flush with large volumes of water. Always wear safety glasses or a shield for eye protection, protective clothing, and observe proper mixing when working with these reagents.

5.2 Aroclors have been tentatively classified as known or suspected human or mammalian carcinogens. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 EQUIPMENT AND SUPPLIES

Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using equipment and supplies other than those specified here, but demonstration of equivalent performance meeting the requirements of this Statement of Work is the responsibility of the Contractor. The Contractor must document in the SDG Narrative when it uses equipment and supplies other than those specified here.

6.1 Glassware

6.1.1 Continuous Liquid-Liquid Extractors - equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication (Hershberg-Wolf extractor, Ace Glass Company, Vineland, NJ P/N 6841-10 or equivalent) or Hydrophobic Membrane-based Extractor (Accelerated One Step™ Extractor, Corning series 3195 or equivalent).

6.1.2 Separatory Funnels - 2 L with Teflon stopcock.

6.1.3 Beakers - 400 ml.

6.1.4 Erlenmeyer Flasks - 250 ml.

6.1.5 Syringes - 1 ml, 2 ml or 10 ml with Luerlok fitting.

6.1.6 Vials and Caps - 20 ml and 10 ml (optional) with screw cap and Teflon or aluminum foil liner, 2 ml capacity for GC auto sampler.

6.1.7 Pipets - 1 ml or 2 ml glass volumetric.

6.1.8 Centrifuge Tube - 12 to 15 ml with 19 mm ground glass joint (optional).

6.1.9 Graduated Cylinder - 1 L capacity.

6.1.10 Drying Column - chromatographic column approximately 400 mm long x 19 mm ID, with coarse frit. (Substitution of a small pad of disposable Pyrex glass wool for the frit will help prevent cross-contamination of sample extracts).

6.1.11 Volumetric Flasks - 1 ml, 2 ml or 10 ml.

6.1.12 Bottle or Test Tube - 20 ml with Teflon-lined screw cap for sulfur removal.

6.1.13 Powder Funnels - 10 cm diameter, for filtration/drying.

6.1.14 Buchner Funnels - 9 cm diameter, for filtration.

6.2 Kuderna-Danish (K-D) Apparatus.

6.2.1 Concentrator Tubes - 15 ml and 10 ml graduated (Kontes K-570050-1025 or K-570040-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground-glass stoppers are used to prevent evaporation of extracts.

6.2.2 Evaporative Flasks - 500 ml (Kontes K-470001-0500, or equivalent). Attach to concentrator tube with springs.

6.2.3 Snyder Column - three-ball macro (Kontes K-503000-0121, or equivalent).

6.2.4 Snyder Column - two-ball micro (Kontes K-569001 -0219, or equivalent).

6.3 pH Paper - wide range (Hydrion Papers, Micro-essential Laboratory, Brooklyn, NY, or equivalent).

6.4 Spatula - stainless steel or Teflon.

6.5 Centrifuge - table top (optional).

6.6 Balances - analytical, capable of accurately weighing ± 0.0001 g, and a top-loading balance capable of weighing 100 g ± 0.01 g. The balances must be calibrated in accordance with ASTM E 617 specifications each 12-hour work shift. The balances must also be annually checked by a certified technician.

6.7 Ultrasonic Cell Disruptor - Heat Systems, Ultrasonics, Inc., Model W-385 (475 watt with pulsing capability, No. 207 3/4-inch tapered disruptor horn) or equivalent device with a minimum 375 watt output capability. NOTE: In order to ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. Erosion of the tip is evidenced by a rough surface.

6.8 Sonabox Acoustic Enclosure (or equivalent) - for use with disruptor to decrease noise level.

6.9 Ultrasonic Water bath

6.10 Filter Paper - No. 41 Whatmann (or equivalent), 9 cm circles (optional).

6.11 Pyrex Glass Wool - rinsed with methylene chloride and dried before use.

6.12 Boiling chips

6.12.1 Silicon carbide boiling chips - approximately 10 to 40 mesh. Heat the chips to 400 °C for 30 minutes or solvent rinse with methylene chloride before use.

6.12.2 Teflon boiling chips (optional) - Rinse with methylene chloride prior to use.

6.13 Water Bath - heated, with concentric ring cover, capable of temperature control. NOTE: The water bath should be used in a hood.

6.14 Nitrogen Evaporation Device - equipped with a heated bath that can be maintained at 35 to 40 °C (N-Evap by Organomation Associates, Inc., South Berlin, MA, or equivalent). To prevent the release of solvent fumes into the laboratory, the nitrogen evaporator device must be used in a hood.

6.15 Oven - drying.

6.16 Desiccator.

6.17 Crucibles - porcelain crucibles or aluminum weighing pans.

6.18 pH Meter - with a combination glass electrode. Calibrate according to manufacturer's instructions. pH meter must be calibrated prior to each use.

6.19 Magnetic Stirrer Motor - Model PC 353, Corning Co., Corning, NY, or equivalent.

6.20 Magnetic Stirrer Bar - Teflon coated, at least 4 cm long.

6.21 Gas Chromatograph/Electron Capture Detector (GC/ECD) System.

6.21.1 Gas Chromatograph - The gas chromatograph (GC) system must be capable of temperature programming and must maintain an optimal flow rate throughout the GC temperature program operations. The system must be suitable for splitless injection and have all required accessories including syringes, analytical columns, and gases.

6.21.2 GC Capillary columns - Two fused silica GC columns are required. A separate electron capture detector is required for each column. The specified analytical columns are a 30 meter DB-1701 (J&W Scientific); SPB 1701 (Supelco); AT 1701 (Alltech); RTX-1701 (Restek); CP-Sil 19CB (Chrompack); 007-1701 (Quadrex); BP-10 (SGE); or equivalent, and a 30 meter DB-608 (J&W Scientific); HP-608 (Hewlett Packard); SPB-608 (Supelco); 007-608 (Quadrex); BP-608 (SGE); CP-Sil 8CB (Chrompack); or equivalent. Note that 30 meters is a minimum requirement for column length. Longer columns may be used as long as they meet all method technical acceptance criteria.

6.21.2.1 The Contractor may choose to use alternate capillary column(s). However, the alternate capillary column(s) selected must meet all the method technical acceptance criteria established in the SOW.

- The GC column must not introduce contaminants which interfere with identification and quantitation of the target compounds listed in Exhibit C (Aroclors).
- The GC column must be able to accept concentrations up to the high point standard for each Aroclor without becoming overloaded.
- The column pair chosen must have dissimilar phases/chemical properties in order to give distinctive chromatograms of each Aroclor.
- The alternate GC column(s) must be used for the entire analysis, including the MDL study, initial and continuing calibration, initial calibration verification and all blank, QC sample and all sample analyses. If a new alternate GC column is chosen after the initial MDL study has been completed, then the MDL study must be reanalyzed using that alternate column. Analytical results generated using any alternate column must meet all technical acceptance criteria listed in the SOW and the CRQLs listed in Exhibit C (Aroclors).

6.21.2.2 The alternate GC column must be designed to optimize performance. Follow manufacturer's instructions for the use of its product. Before use of any column, other than the ones specified in 6.21.2, the Contractor must meet the criteria listed in 6.21.2.1. Once this has been demonstrated, the Contractor must document the column used (brand name, length, diameter, and film thickness) in each SDG Narrative.

6.21.2.3 Manufacturer provided technical information concerning the performance characteristics of the GC column(s) must be included in the MDL Study data package to support the use of any alternate column.

6.21.2.4 A description of the GC columns used for the analysis shall be provided in the SDG Narrative.

6.21.2.5 Packed columns must not be used.

6.21.2.6 Columns are mounted in a 0.25-inch injector ports by using glass adapters available from a variety of commercial sources (J&W Scientific, Supelco, Inc., Hewlett-Packard, Varian, Inc., Perkin Elmer, or equivalent). The two columns may be mounted into a single injection port with a tee adapter (Supelco, Inc., Bellefonte, PA, Catalog No. 2-3660, or equivalent). Use of this adapter allows simultaneous injection onto both columns. The laboratory should follow manufacturer's recommendations for mounting capillary columns in injector ports.

6.21.2.7 The carrier gas for routine applications is helium. Laboratories may choose to use hydrogen as a carrier gas, but they must clearly identify its use in the SDG Narrative and on all divider pages preceding raw chromatographic data in submissions to the Agency. Laboratories that choose to use hydrogen are advised to exercise caution in its use. Use of a hydrogen leak detector is highly recommended when hydrogen is used as the carrier gas. All GC carrier gas lines must be constructed from stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants or flow controllers with rubber components are not to be used.

6.21.3 Electron Capture Detector (ECD) - the linearity of the response of the ECD may be greatly dependent on the flow rate of the make-up gas. The make-up gas must be P-5, P-10 (argon/methane) or nitrogen according to the instrument specification. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector. The GC/ECD system must be in a room in which the atmosphere has been demonstrated to be free of all contaminants which may interfere with

the analysis. The instrument must be vented to outside the facility or to a trapping system which prevents the release of contaminants into the instrument room.

6.21.4 Data System - a data system must be interfaced to the GC/ECD. The data system must allow the continuous acquisition of data throughout the duration of the chromatographic program and must permit, at the minimum, the output of time vs. intensity (peak height or peak area) data. Also, the data system must be able to rescale chromatographic data in order to report chromatograms meeting the requirements listed within this method.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Reagent water - defined as water in which no interferant is observed at one-half the CRQL of any target Aroclor. Reagent water may be generated by passing tap water through a carbon filter bed containing about 453 g of activated carbon (Calgon Corp., Filtrasorb - 300 or equivalent).

7.1.2 Sodium sulfate - granular-anhydrous reagent grade, heated at 400 °C for 4 hours, or at 120 °C for 16 hours, cooled in a desiccator, and stored in a glass bottle (Baker anhydrous granular, Catalog No. 3375, or equivalent). Each lot must be extracted with hexane and analyzed by GC/ECD to demonstrate that it is free of interference before use. CAUTION: An open container of sodium sulfate may become contaminated during storage in the laboratory.

7.1.3 Concentrated sulfuric acid (H_2SO_4) - 18 N.

7.1.4 Sulfuric acid/water (H_2SO_4/H_2O), 1:1, v/v.

7.1.5 Sodium hydroxide solution (NaOH) (10 N) - carefully dissolve 40 g of NaOH in reagent water and dilute the solution to 100 ml.

7.1.6 Methylene chloride, hexane, acetone, toluene, iso-octane, and methanol (optional) - pesticide quality or equivalent. It is recommended that each lot of solvent used be analyzed to demonstrate that it is free of interference before use. Methylene chloride must be certified as acid free or must be tested to demonstrate that it is free of hydrochloric acid. Acidic methylene chloride must be passed through basic alumina and then demonstrated to be free of hydrochloric acid.

7.1.7 Mercury - triple distilled, for sulfur cleanup.

7.1.8 Copper powder (optional) - fine, granular (Mallinckrodt 4649 or equivalent). Copper may be used instead of mercury for sulfur cleanup. Remove oxides by treating with dilute nitric acid, rinse with distilled water to remove all traces of acid, rinse with acetone, and dry under a steady stream of nitrogen.

7.2 Standards

7.2.1 Standards Documentation

The Contractor must provide all standards to be used with this contract. These standards may be used only after they have been certified according to the procedure described in Exhibit E. The Contractor must be able to verify that the standards are certified by producing the manufacturer's certificates and/or generating the documentation as described in Exhibit E. Manufacturer's certificates of analysis must be retained by the Contractor for the term of the contract. The documentation may be requested during an on-site audit.

7.2.2 Stock standard solutions (1 µg/µL) - can be prepared from pure reference standard materials or purchased as certified solutions.

7.2.2.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in hexane or iso-octane and dilute to volume in a 10 ml volumetric flask. Larger volumes may be used at the convenience of the analyst.

7.2.2.2 When compound purity is assayed to be 97 percent or greater, the weight may be used without correction to calculate the concentration of the stock solution. If the compound purity is assayed to be less than 97 percent, the weight must be corrected when calculating the concentration of the stock solution. See Exhibit E for Analytical Standards Requirements.

7.2.2.3 Fresh stock standards must be prepared once every twelve months or sooner, if standards have degraded or concentrated. Stock

standards must be checked for signs of degradation or concentration just prior to preparing working standards from them.

7.2.3 Secondary Dilution Standards

7.2.3.1 Using stock standards, prepare individual secondary dilution standards in hexane or iso-octane that contain each Aroclor of interest at 10 µg/ml, except for Aroclor 1221, which is prepared at 20 µg/ml.

7.2.3.2 Fresh secondary dilution standards must be prepared once every six months after the preparation date (or the date opened for purchased standards). The standards must be replaced sooner if the standard has demonstrated signs of degradation or evaporation.

7.2.4 Working Standards

7.2.4.1 Surrogate Standard Spiking Solution

7.2.4.1.1 The surrogates, tetrachloro-m-xylene and decachlorobiphenyl, are added to all standards; aqueous samples, soil/sediment/solid samples; QC samples and blanks. Prepare a surrogate standard spiking solution containing each of the surrogates at 0.2 µg/ml in hexane or iso-octane. Prepare fresh surrogate standard spiking solution monthly, or sooner if the solution has degraded or evaporated.

7.2.4.1.2 For oil and oily sludge samples extracted using the waste dilution procedure described in Section 10.1.5, prepare a surrogate spiking solution containing the surrogates, tetrachloro-m-xylene and decachlorobiphenyl, at five (5) times the concentration specified above (1 µg/ml in hexane). Prepare fresh surrogate standard spiking solution monthly, or sooner if the solution has degraded or evaporated.

7.2.4.2 Matrix Spiking Solution

7.2.4.2.1 Prepare a matrix spiking solution in hexane or iso-octane that contains Aroclor 1254 at a concentration of 0.1 µg/ml. Aroclor 1254 must be added to all designated MS/MSD samples unless instructed otherwise by the RSCC. In specific cases where site history, previous sampling or sample screening indicates the presence of a known Aroclor other than 1254, that specific Aroclor may be designated by the Region as the matrix spiking compound. The concentration of the designated Aroclor in the matrix spiking solution shall remain at 0.1 µg/ml (0.2 µg/ml for Aroclor 1221).

7.2.4.2.2 For oil and oily sludge samples extracted using the waste dilution procedure described in Section 10.1.5, prepare a matrix spiking solution of the appropriate Aroclor at five (5) times the concentration specified above (0.5 µg/ml in acetone/1.0 µg/ml for Aroclor 1221). Prepare fresh matrix spiking solution monthly, or sooner if the solution has degraded or evaporated.

7.2.4.3 Initial Calibration Standards

This method is geared to analyze for PCBs only in all matrices. If the site specific Aroclor(s) are known at the onset of the sampling event, the site specific Aroclor will be specified to the Contractor either by the RSCC or on the chain of custody form accompanying each sample delivery group. In this case, the Contractor must prepare and analyze a three point calibration curve of the site specific Aroclor and one concentration level of each of the other individual Aroclor standards. If the nature of the site is unknown, then the Contractor must prepare and analyze a three point calibration curve of Aroclor 1254 to demonstrate the linearity of the GC/ECD and one concentration level of each of the other Aroclor standards in order to identify any unknown Aroclors.

For the target Aroclor (or Aroclor 1254, if unknown) requiring a three point calibration curve, the low point concentration must correspond to the CRQL. The mid point concentration must be 4 times the low point concentration and the high point concentration must be at least 10 times the low point concentration. A higher concentration may be chosen by the Contractor. The high point concentration defines the upper range for which the calibration is valid. The surrogates are also included at three concentration levels in these initial calibration standards for the purposes of calibration.

Individual Aroclor standards of each of the other Aroclors are prepared at a single concentration level which must be equal to the mid point concentration level of the calibration curve (4 times the CRQL). Each individual Aroclor standard must contain both surrogates at a concentration of 20 ng/ml.

	Concentration (ng/ml)		
Aroclor	(low)	(mid)	(high)
1016/1260		400	
1221		800	
1232		400	
1242		400	
1248		400	
1254*	100	400	1000
1262		400	
1268		400	
TCX	5.0	20	50
DCB	5.0	20	50

* = Site specific Aroclor may be substituted
TCX = Tetrachloro-m-xylene (surrogate)
DCB = Decachlorbiphenyl (surrogate)

If additional Aroclors are also known or found to be present, an initial calibration curve of each of the additional Aroclors must also be prepared and analyzed within each 72 hour analytical sequence.

7.2.4.4 Continuing Calibration Standards

Prepare a continuing calibration standard of the target Aroclor (or Aroclor 1254, if unknown) which was calibrated as specified above, at the midpoint concentration of 400 ng/ml (800 ng/ml for Aroclor 1221) in hexane or iso-octane and include the surrogates, TCX and DCB, at 20 ng/ml.

In cases where the site history, previous sampling, or sample screening indicates the presence of a known Aroclor, and the GC/ECD was calibrated using that specific Aroclor; that Aroclor must also be used as the continuing calibration standard. If additional Aroclors are also known or found to be present during sample analysis, a continuing calibration standard for each of the

additional Aroclors must also be prepared and analyzed.

7.2.5 Ampulated Standard Extracts

Standard solutions purchased from a chemical supply house as ampulated extracts in glass vials may be retained for 2 years from the manufacturer's preparation date, unless the manufacturer recommends a shorter time period. Standard solutions prepared by the Contractor which are immediately ampulated in glass vials may be retained for 2 years from the preparation date. Upon breaking the glass seal, the expiration times listed in applicable standard preparation sections will apply. The Contractor is responsible for assuring that the integrity of the standards have not degraded by following proper storage procedures (see Section 7.3).

7.3 Storage of Standard Solutions

7.3.1 Store the stock and secondary dilution standard solutions at 4 °C (± 2 °C) in Teflon-lined screw cap amber bottles/vials. Fresh standards should be replaced or prepared every twelve months at a minimum.

7.3.2 Store all other working standard solutions in amber glass bottles or vials with Teflon lined screw caps at 4 °C (± 2 °C) and protect from light. The standard solutions must be checked frequently for stability. Replace all working standard solutions after six months, or sooner if comparison with quality control check samples indicates a problem. CAUTION: Analysts must allow all standard solutions to equilibrate to room temperature before use.

7.3.3 Protect all standards from light. Samples, sample extracts and standards must be stored separately.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 Sample Collection and Preservation

8.1.1 Water samples may be collected in 1 L amber glass containers, fitted with screw-caps lined with Teflon. If amber containers are not available, the samples should be protected from light. Soil samples may be collected in glass containers or closed end tubes (e.g., brass sleeves) in sufficient quantity to perform the analysis. The specific requirements for site sample collection are outlined by the Region.

8.1.2 If samples are received in containers other than glass, then the Contractor shall contact the RSCC to ascertain the proper procedure for subsampling from the sample container.

8.1.3 All samples must be iced and/or refrigerated at 4 °C (±2 °C) from the time of collection until extraction.

8.2 Procedure for Sample Storage

8.2.1 The samples must be protected from light and refrigerated at 4 °C (±2 °C) from the time of receipt until 60 days after delivery of a complete reconciled sample data package to the Agency. After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

8.2.2 If sample storage temperatures exceed 4°C (±2 °C) and/or samples are not light protected, then the Contractor shall contact the RSCC to ascertain whether or not the samples should be analyzed. For all samples that were not properly refrigerated and/or light protected, the Contractor shall note the problem, the EPA sample numbers for the affected samples, and the Regional instructions in the SDG Narrative.

8.2.3 The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.3 Procedure for Sample Extract Storage

8.3.1 Sample extracts must be protected from light and stored at 4°C (±2 °C) until 365 days after delivery of a complete reconciled data package to the Agency.

8.3.2 Sample extracts must be stored in an atmosphere demonstrated to be free of all potential contaminants.

8.3.3 Samples, sample extracts, and standards must be stored separately.

8.4 Contract Required Holding Times

8.4.1 Extraction of water samples by separatory funnel procedures must be completed within five days of the Validated Time of Sample Receipt (VTSR). Extraction of water samples by continuous liquid-liquid extraction procedures must be started within five days of VTSR. Extraction of soil/sediment/solid samples by sonication must be completed within 10 days of VTSR. Oil and oily sludge (waste) dilutions must be completed within 10 days of VTSR.

8.4.2 As part of the Agency's QA program, the Agency may provide Performance Evaluation (PE) samples as standard extracts which the Contractor is required to prepare per instructions provided by the Agency. The extraction holding times (five days after VTSR for water, 10 days after VTSR for soil/sediment/solid and oil/oily sludge) do not apply for PEs received as standard extracts. The PE samples must be analyzed and reported with the SDG with which they were submitted.

8.4.3 Extracts of water, soil/sediment/solid or oil/oily sludge must be analyzed within 40 days of the start of extraction.

8.4.4 If samples submitted for PCB analysis have exceeded contract required holding times and have not yet been extracted and/or analyzed, then the Contractor shall contact the RSCC to ascertain whether or not the

samples should be extracted and/or analyzed. Note that this notification requirement in no way obviates the contractual requirement for the Contractor to extract and/or analyze samples within holding times. If the Contractor is instructed to proceed with extraction and/or analysis outside holding times, sample price may be reduced depending upon the impact of the non-compliance on data usability. For all samples that exceeded holding times, the Contractor shall note the problem, the EPA sample numbers for the affected samples, and the Regional instructions in the SDG narrative.

8.4.5 PCB data reported from sample preparation and/or analyses which were performed outside the contract required holding times for extraction and/or analysis shall be subject to a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Gas Chromatograph Operating Conditions

9.1.1 The following are the gas chromatographic analytical conditions. The conditions are recommended unless otherwise noted.

Carrier Gas:	Helium (hydrogen may be used, see 6.21.2.7)
Column Flow:	30-45 cm/sec (helium)
Make-up Gas:	P-5/P-10 or N_2 (required)
Injector Temperature:	> 200 °C (see Section 9.1.4)
Injection:	On-column
Injection Volume:	1 or 2 µL (see Section 9.1.3)
Injector:	Grob-type, splitless
Initial Temperature:	150 °C
Initial Hold Time:	½ min
Temperature Ramp:	5 C° to 6 C°/min
Final Temperature:	275 °C
Final Hold Time:	Until after decachlorobiphenyl has eluted (approximately 10 minutes)

9.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, the same GC conditions must be used for the analysis of all standards, samples, blanks and QC samples. The linearity of the ECD may be greatly dependent on the flow rate of the make-up gas. Initial make-up flow testing, using a single Aroclor standard at the 3 concentrations (low, mid, and high), may be required to determine the optimum flow rate for the best linearity for the detector. Care must be taken to maintain stable and appropriate flow of make-up gas to the detector.

9.1.3 Manual injections must be 2 µL. Autosamplers may use 1 µL volumes. The same injection volume must be used for all standards, blanks, and samples, including QC samples.

9.1.4 Cold (ambient temperature) on-column injectors that allow injection directly onto a column may be used as long as the MDL study, initial calibration and continuing calibration technical acceptance criteria are met.

9.2 Initial Calibration

9.2.1 Summary of Initial Calibration

This method is geared to analyze for PCBs only in all matrices. If the site specific Aroclor(s) are known at the onset of the sampling event, the site specific Aroclor will be specified to the Contractor either by the RSCC or on the chain of custody form accompanying each sample delivery group. In this case, the Contractor must calibrate the GC/ECD system following the procedures below using the site specific Aroclor to calibrate the instrument and one concentration level of each of the other individual Aroclor standards for identification purposes. If the nature of the site is unknown then the calibration must include a three point calibration curve of Aroclor 1254 to demonstrate the linearity of the GC/ECD as well as analysis of at one concentration level of each of the other Aroclor standards. The concentrations of Aroclors in the initial calibration sequence are specified in Section 7.2.4.3. If additional Aroclors are identified during the analytical sequence, the Contractor must perform a three point initial calibration curve of each of the additional Aroclors within 72 hours of the first detection of that Aroclor in any sample analytical sequence.

9.2.2 Frequency of Initial Calibration

Each GC/ECD system must be initially calibrated upon award of the contract; when the Contractor is informed of a site specific target Aroclor which requires initial calibration; when an Aroclor is

identified in an unknown sample during the analytical sequence; whenever major instrument maintenance or modification is performed (e.g., column replacement or repair, cleaning or replacement of ECD, etc.) or if the initial calibration and/or continuing calibration technical acceptance criteria have not been met.

9.2.3 Procedure for Initial Calibration

9.2.3.1 Set up the GC/ECD system as described in Section 9.1

9.2.3.2 Prepare the initial calibration standard(s) using the Aroclors and the concentrations specified in Section 7.2.4.3.

9.2.3.3 All standards, samples, QC samples, blanks and extracts must be allowed to warm to ambient temperature before analysis.

9.2.3.4 The initial calibration analytical sequence is presented below. At least one three point calibration curve is analyzed with each analytical sequence in order to demonstrate instrument linearity. At a minimum, the calibration will include a three point calibration curve of the target Aroclor (or Aroclor 1254, if unknown) to demonstrate the linearity of the GC/ECD as well as analysis of one concentration level of each of the other individual Aroclor standards. NOTE: The final two steps of the initial calibration sequence, with the injection of the instrument blank and the continuing calibration standard, mark the beginning of the first 12-hour analytical sequence in which samples may be run.

9.2.3.4.1 The initial calibration sequence is summarized below:

INITIAL CALIBRATION SEQUENCE

1.	Target Aroclor (or 1254)	low
2.	Target Aroclor (or 1254)	mid
3.	Target Aroclor (or 1254)	high
4.	AR1016/1260	mid
5.	AR1221	mid
6.	AR1232	mid
7.	AR1242	mid
8.	AR1248	mid
9.	AR1254 *	mid
9.	AR1262	mid
10.	AR1268	mid
11.	Instrument Blank	
12.	Continuing Calibration Standard/Target Aroclor (or AR1254)	mid
13 etc...	Samples	

If the analytical batch includes samples where the target Aroclor is known, as well as samples where the target Aroclor is unknown, then the calibration sequence presented above must be used but the three point curve must be of the known Aroclor. (e.g., if AR 1262 is identified as the target PCB, run a three point curve of AR 1262 and the mid point of AR 1254). In cases where samples from various sites are analyzed and several target Aroclors are expected, it will be necessary to include three point initial calibration curves for each expected Aroclor. In such cases, a continuing calibration standard for each of the expected Aroclors must also be included in the analytical sequence at the required frequency specified for continuing calibrations in Section 9.3.2.

9.2.3.5 A 3-point initial calibration curve (Section 7.2.4.3) must be analyzed for each Aroclor found to be present in the samples to assure linearity of response. If additional Aroclors are identified during the analytical sequence, the Contractor must perform a three point initial calibration curve of each of the additional Aroclors within 72 hours of the first detection of that Aroclor in any sample analytical sequence.

9.2.4 Calculations for Initial Calibration

9.2.4.1 During the initial calibration sequence, absolute retention times (RT) are determined for the major peaks (3 to 5) for each Aroclor and for the surrogates in all calibrations standards. Calculate a mean absolute retention time for the Aroclors and surrogate compounds which were calibrated using a three point curve. Determine the absolute retention times for each individual Aroclor analyzed at one level.

9.2.4.2 Retention time windows are calculated for the major peaks (3 to 5) of each Aroclor and for the surrogates using the criteria listed in Table 1. Windows are centered around the absolute retention times established above (Section 9.2.4.1). Aroclors are identified when peaks are observed in each of the RT windows for the 3 to 5 major peaks on both GC columns.

9.2.4.3 The linearity of the instrument is determined by calculating a percent relative standard deviation (%RSD) of the calibration factors for the three-point calibration curve for at least one Aroclor and the surrogates. If more than one calibration curve for any Aroclor was analyzed at any point in the analytical sequence, the %RSD for each curve must be calculated and reported. Either peak area or peak height may be used to calculate the calibration factors used in the %RSD equation. For example, it is permitted to calculate linearity for Aroclor 1254 based on peak area and to calculate linearity for Aroclor 1248 based on peak height. It is not permitted within a %RSD calculation for an Aroclor to use calibration factors calculated from both peak area and peak height. For example, it is not permitted to calculate the calibration factor for the low point standard for Aroclor 1254 using peak height and calculate the midpoint and high point standard calibration factors for Aroclor 1254 using peak area.

9.2.4.4 Calculate the calibration factor (CF) for each peak in a selected set of three to five major peaks for each Aroclor standard over the initial calibration curve using Equation 1. The Contractor must choose different peaks to quantitate each Aroclor. Any peak common to more than one Aroclor must not be used to quantitate either compound. Calculate the calibration factors for the surrogates in the initial calibration using Equation 1. Also calculate the calibration factor for each peak in a selected set of three to five major peaks for each individual Aroclor analyzed at one level using Equation 1.

$$CF = \frac{\textit{Peak area (or height) of the standard}}{\textit{Mass injected (ng)}}$$

EQ. 1

9.2.4.5 Calculate the mean calibration factors for each Aroclor/surrogate calibration curve and the %RSD of the calibration factors using Equations 2 and 3.

EQ. 2

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

EQ. 3

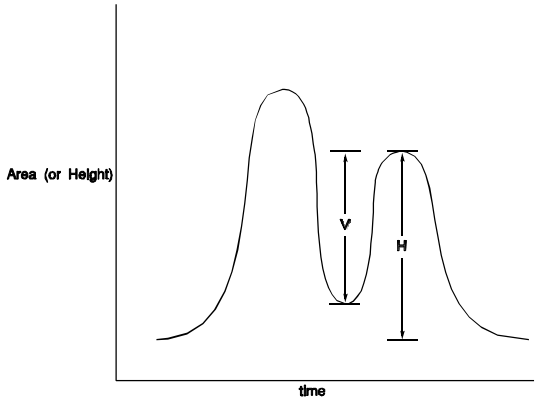
$$\%RSD = \frac{SD_{CF}}{\overline{CF}} \times 100$$

Where,

$$SD_{CF} = \sqrt{\frac{\sum_{i=1}^{n} (CF_i - \overline{CF})^2}{(n-1)}}$$

$\overline{CF}$	=	Mean calibration factor
%RSD	=	Percent relative standard deviation
SD_{CF}	=	Standard deviation of calibration factors
CF_i	=	Individual Calibration factors
n	=	Total number of values (3)

9.2.4.6 Calculate the resolution between each of the Aroclor peaks selected for quantitation in each of the mid point Aroclor standards using Equation 4.



EQ.4

$$\%Resolution = \frac{V}{H} \times 100$$

Where,

V = Depth of the valley between the two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks.

H = Height of the shorter of the adjacent peaks, measured from the baseline.

9.2.5 Technical Acceptance Criteria for Initial Calibration

All initial calibration technical acceptance criteria apply independently to both GC columns.

9.2.5.1 The initial calibration sequence must be analyzed according to the procedure (and in the order listed) in Section 9.2.3.4, at the concentrations listed in Sections 7.2.4.3, and at the frequency listed in Section 9.2.2. The GC/ECD operating conditions optimized in Section 9.1 must be followed.

9.2.5.2 The %RSD of the calibration factors for any Aroclor target compound which was calibrated using a 3-point calibration curve must be less than or equal to 20.0 percent. The %RSD of the calibration factors for the surrogates must be less than or equal to 30.0 percent.

9.2.5.3 All required blanks must meet the technical acceptance criteria in Section 12.1.

9.2.5.4 The resolution between each of the Aroclor peaks selected for quantitation must be greater than or equal to 60 percent ($\geq$ 60 %).

9.2.5.5 The identification of multicomponent analytes by gas chromatographic methods is based primarily on pattern recognition and on peak retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented.

- The chromatograms of the standards for the Aroclors analyzed during the initial calibration sequence must display all peaks chosen for identification of each analyte at greater than 25 percent and less than 100 percent of full scale.
- If a chromatogram is replotted electronically to meet the technical acceptance criteria, the scaling factor used must be displayed on the chromatogram.
- If the chromatogram of any standard needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

9.2.6 Corrective Action for Initial Calibration

9.2.6.1 If the technical acceptance criteria for the initial calibration are not met, inspect the system for problems. It may be necessary to change the column, bake out the detector, clean the injection port, or take other corrective actions to achieve the technical acceptance criteria.

9.2.6.2 Contamination should be suspected as a cause if the detector cannot achieve acceptable linearity using this method. In the case of low level contamination, baking out the detector at an elevated temperature (350 °C) should be sufficient to achieve acceptable performance. In the case of heavy contamination, passing hydrogen through the detector for 1-2 hours at an elevated temperature may correct the problem. In the case of severe contamination, the detector may require servicing by the ECD manufacturer. DO NOT OPEN THE DETECTOR. THE ECD CONTAINS RADIOCHEMICAL SOURCES.

9.2.6.3 If a laboratory cleans out a detector using an elevated temperature, the ECD electronics must be turned off during the bake out procedure. Follow the manufacturers instructions describing the bake out procedure.

9.2.6.4 After bake out or hydrogen reduction, the detector must be recalibrated using the initial calibration sequence (Section 9.2).

9.2.6.5 Initial calibration technical acceptance criteria must be met before any samples, including QC samples or required blanks are analyzed. Any samples or required blanks analyzed before the initial calibration technical acceptance criteria have been met will require reanalysis at no additional cost to the Agency. Reanalyses must be performed within contract required holding times and must meet all sample technical acceptance criteria.

9.2.6.6 Sample analyses reported with a non-compliant initial calibration after reanalysis shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

9.3 Continuing Calibration

9.3.1 Summary of Continuing Calibration

The GC system is checked every twelve hours by the analysis of an instrument blank and continuing calibration check standards. The analysis of continuing calibration check standards ensures that the GC system continues to meet instrument sensitivity and linearity requirements of the SOW. Sample data are not acceptable unless bracketed by acceptable analyses of instrument blanks and continuing calibration check standards. Continuing calibration check standards must be included for all target Aroclors which were initially calibrated with a three point calibration curve. Continuing calibration check standards must also be included at the designated frequency for all Aroclors which were identified during the analytical sequence.

9.3.2 Frequency of Continuing Calibration

The initial calibration must be checked at the end of the initial calibration sequence (Section 9.2.3.4.1) and every 12 hours with the analysis of an instrument blank and the midpoint of each target Aroclor standard or standards. The 12 hour time requirement is a minimum; more frequent analyses may be advantageous to continue a sequence.

9.3.2.1 The final two steps of the initial calibration sequence, with the injection of the instrument blank and continuing calibration check standards, mark the beginning of the first 12-hour analytical sequence in which samples may be run. Samples may be injected for the remainder of the 12-hour period following the injections of the instrument blank and continuing calibration check standards. The injections immediately after that 12-hour period must be an instrument blank and continuing calibration check standards. The instrument blank must be analyzed first, before the standards.

9.3.2.2 The analyses of the instrument blank and continuing calibration check standards immediately following one 12-hour period may be used to begin the subsequent 12-hour period, provided that they meet the technical acceptance criteria in Section 9.3.5. In that instance, the subsequent 12-hour period must be bracketed by the acceptable analyses of an instrument blank and continuing calibration check standards, in that order. Those two analyses may in turn be used to bracket the front end of yet another 12-hour period. This progression may continue every 12 hours until such time as any of the instrument blanks or continuing calibration check standards fail to meet the technical acceptance criteria in Section 9.3.5, or 72 hours have elapsed, whichever comes first. Successive 12-hour time periods begin with the injection of the instrument blank.

9.3.2.3 Instrument blanks and a continuing calibration check standard or standards must be analyzed every twelve hours or at the end of an analytical run, whichever is more frequent.

9.3.2.4 If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an acceptable instrument blank and the midpoint continuing calibration standards of each Aroclor must be analyzed in order to start a new sequence. This requirement applies even if no analyses were performed since the standards were injected.

9.3.2.5 If the entire 12-hour period is not required for the analyses of all samples to be reported, the sequence must be ended with the instrument blank/continuing calibration standard(s) combination.

9.3.2.6 If an additional Aroclors are identified in any sample during the analytical sequence, a 3-point initial calibration curve for that Aroclor must be run within 72 hours of that detection (from time of injection), and within a valid 12-hour analytical sequence.

9.3.3 Procedure for Continuing Calibration

9.3.3.1 Analyze the instrument blank and continuing calibration check standard or standards at the required frequencies (Section 9.3.2). The concentration of continuing calibration check standards shall be the midpoint of the initial calibration standards and shall be prepared from the same standard solution.

9.3.3.2 Continuing calibration standards must be analyzed for each target Aroclor (or Aroclor 1254, if PCBs are unknown or not detected) which was included in the initial calibration curve. If more than one Aroclor was initially calibrated over a three point curve, the Contractor must analyze continuing calibration standards for all target Aroclors. If additional Aroclors are identified during the analytical sequence (Section 9.2.3) and are subsequently calibrated, continuing calibration standards of that Aroclor must be included in every 12 hour sequence.

9.3.3.3 All standards and blanks must be at ambient temperature at the time of preparation and analysis.

9.3.4 Calculations for Continuing Calibration

9.3.4.1 Calculate calibration factors (CF) for the surrogate compounds and for each peak in the set of peaks for the target Aroclor or Aroclors in all continuing calibration check standards using Equation 1.

9.3.4.2 Calculate the percent difference (%D) between the continuing calibration check standard CF and the associated initial calibration mean calibration factor (Section 9.2.4.4) for each target Aroclor and the surrogate compounds using Equation 5.

EQ. 5

$$\%Difference = \frac{CF_c - \overline{CF_i}}{\overline{CF_i}} \times 100$$

Where,

CF_c = Calibration factor from continuing calibration standard

$\overline{CF_i}$ = Mean calibration factor from the associated initial calibration meeting technical acceptance criteria

9.3.4.2 If additional Aroclors are identified during the analytical sequence (Section 9.2.3), the Contractor must perform a three point calibration curve for that Aroclor within 72 hours of the first detection in any sample. Any three point calibration analyzed within an analytical sequence must meet all technical acceptance criteria for initial calibration as defined in Section 9.2.5. The percent difference between the three point calibration and the individual Aroclor, which was included in the initial calibration sequence, must also be calculated using the above equation.

9.3.5 Technical Acceptance Criteria for Continuing Calibration

All continuing calibration technical acceptance criteria apply independently to each GC column. Each GC column must meet criteria.

9.3.5.1 The continuing calibration standard(s) and instrument blanks must be analyzed at the required frequency on a GC/ECD system that has met the initial calibration technical acceptance criteria.

9.3.5.2 The absolute retention time for each Aroclor peak (in the set of 3 to 5 major peaks) and surrogates in the continuing calibration standards must be within the retention time window determined from the initial calibration (Section 9.2.4.2) .

9.3.5.3 The % Difference (%D) between the calibration factors for each target Aroclor must be greater than or equal to -25 percent and less than or equal to 25 percent. The %D of the calibration factor for the surrogate compounds must be within the inclusive range of ± 30 percent. One target compound (not the surrogate) per column may exceed the 25 percent limit but that compound must be within the inclusive range of 35 percent.

9.3.5.4 All required blanks must meet the technical acceptance criteria in Section 12.1.

9.3.5.5 The 60 % peak resolution and compound identification technical acceptance criteria defined for the initial calibration (Section 9.2.5.4 and 9.2.5.5.) must also be met for all continuing calibration check standard(s).

9.3.6 Corrective Action for Continuing Calibration

9.3.6.1 If the technical acceptance criteria for the continuing calibration are not met, inspect the system for problems and take corrective action to achieve the acceptance criteria.

9.3.6.2 Major corrective actions such as replacing the GC column or baking out the detector will require that a new initial calibration be performed that meets the technical acceptance criteria in Section 9.2.5.

9.3.6.3 Minor corrective actions may not require performing a new initial calibration, provided that a new analysis of the continuing calibration standard that originally failed the criteria and an associated instrument blank immediately after the corrective action do meet all the technical acceptance criteria.

9.3.6.4 If the continuing calibration standard does not meet technical acceptance criteria listed above, it must be reinjected immediately. If the second injection of the continuing calibration check standard meets the criteria, sample analysis may continue. If the second injection does not meet the criteria, all data collection must be stopped. Appropriate corrective action must be taken, and a new initial calibration sequence must be run before more sample data are collected.

9.3.6.5 If an instrument blank does not meet the technical acceptance criteria listed in Section 12.1.4.4, all data collection must be stopped. Appropriate corrective action must be taken to clean out the system, and an acceptable instrument blank must be analyzed before more sample data are collected.

9.3.6.6 Analysts are reminded that running an instrument blank and continuing calibration check standards once every 12 hours is the minimum contract requirement. Late eluting peaks may carry over from one injection to the next if highly complex samples are analyzed or if the GC conditions are unstable. Such carryover is unacceptable. Therefore, it may be necessary to run instrument blanks and continuing calibration standards more often to avoid discarding data.

9.3.6.7 If successful instrument blanks and continuing calibration check standards cannot be run after an interruption in analysis, an acceptable initial calibration must be run before sample data may be collected. All sample analyses in a sample delivery group must be preceded by a compliant initial calibration and followed by compliant instrument blanks and continuing calibration check standards, as described in Section 9.3.2.

9.3.6.8 Continuing calibration check technical acceptance criteria must be met before any samples, including QC samples and required blanks are reported. Any samples, including QC samples and required blanks associated with a continuing calibration check standard which did not meet the technical acceptance criteria will require reanalysis at no additional cost to the Agency. Re-analyses must be performed within contract required holding times and must meet all sample technical acceptance criteria.

9.3.6.9 Sample analyses reported with non-compliant continuing calibration technical acceptance criteria after reanalysis shall receive a commensurate reduction in sample price or zero payment due to data rejection depending upon the impact of the non-compliance on data usability.

10.0 PROCEDURE

10.1 Sample Preparation

10.1.1 If insufficient sample amount (less than 90% of the required amount) is received to perform the analyses, the Contractor shall contact the RSCC to apprise them of the problem. The Region will either require that no sample analyses be performed or will require that a reduced volume be used for the sample analysis. All changes in the analyses **must** be preapproved by the Region I Project Officer. The Contractor shall document the Region's decision (including sample weight/volume prepared and analyzed) in the SDG Narrative.

10.1.2 If multiphase samples (e.g., a two-phase liquid sample, oily sludge/sandy soil sample) are received by the Contractor, the Contractor shall contact the RSCC to apprise them of the type of sample received. If all phases of the sample are amenable to analysis, the Region may require the Contractor to do one of the following:

- Mix the sample and analyze an aliquot from the homogenized sample.
- Separate the phases of the sample and analyze each phase separately. The RSCC will provide EPA sample numbers for the additional phases.
- Separate the phases and analyze one or more of the phases, but not all of the phases. The RSCC will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.1 If all of the phases are not amenable to analysis (i.e., outside the scope of the SOW), then the Region may require the Contractor to do one of the following:

- Separate the phases and analyze the phases that are amenable to analysis. The RSCC will provide EPA sample numbers for the additional phases, if required.
- Do not analyze the sample.

10.1.2.2 No other change in the analyses will be permitted. The Contractor shall document the problem, the EPA sample numbers for the affected samples and the Region's instructions in the SDG Narrative.

10.1.3 Extraction of Water Samples

Water samples may be extracted by either a separatory funnel procedure or a continuous liquid-liquid extraction procedure. If an emulsion prevents acceptable solvent recovery with the separatory funnel procedure, continuous liquid-liquid extraction must be employed.

10.1.3.1 Separatory Funnel Extraction

10.1.3.1.1 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder. Measure and record pH of the sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Record the pH of the sample "as received" and the exact volume used for extraction on the sample preparation log and on the Form I PCB. Samples requiring pH adjustment must be noted in the SDG Narrative. Pour the sample into a 2 L separatory funnel.

10.1.3.1.2 Using a syringe or a volumetric pipet, add 1.0 ml of the surrogate standard spiking solution (0.2 µg/ml/Section 7.2.4.1.1) to all water samples.

10.1.3.1.3 Rinse the graduated cylinder with 30 ml of methylene chloride

and transfer the rinsate to the separatory funnel. Rinse the empty 1 L container with 30 ml of methylene chloride and add the rinsate to the separatory funnel. Extract the sample by shaking the funnel for two minutes, with periodic venting to release excess pressure. NOTE: The total volume of solvent used for extraction is 60 ml. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation or other physical means. Drain the methylene chloride into a 250 ml Erlenmeyer flask.

10.1.3.1.4 Add a second 60 ml volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Proceed to Section 10.2.

10.1.3.2 Continuous Liquid-Liquid Extraction

10.1.3.2.1 Continuous Liquid-Liquid Extraction Without Hydrophobic Membrane

10.1.3.2.1.1 Follow manufacturer's instructions for set-up.

10.1.3.2.1.2 Add methylene chloride to the bottom of the extractor and fill it to a depth of at least one inch above the bottom sidearm.

10.1.3.2.1.3 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder; transfer the aliquot to the continuous extractor. Measure the pH of the sample with wide range pH paper and record pH. Adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, as required. Record the pH of the sample "as received" and the exact volume used for extraction on the sample preparation log and on the Form I PCB. Samples requiring pH adjustment must be noted in the SDG Narrative. NOTE: With some samples, it may be necessary to place a layer of glass wool between the methylene chloride and the water layer in the extractor to prevent precipitation of suspended solids into the methylene chloride during extraction.

10.1.3.2.1.4 Using a syringe or volumetric pipet, add 1.0 ml of the surrogate standard spiking solution (0.2 µg/ml/Section 7.2.4.1.1) into the sample and mix well.

10.1.3.2.1.5 Rinse the graduated cylinder with 30 ml of methylene chloride and transfer the rinsate to the continuous extractor. Rinse the empty 1 L container with 30 ml of methylene chloride and add the rinsate to the continuous extractor.

10.1.3.2.1.6 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 5 to 15 ml/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 18 hours. NOTE: When a minimum drip rate of 10-15 mls/minute is maintained throughout the extraction, the extraction time may be reduced to a minimum of 12 hours. Allow to cool, then detach the distillation flask. Proceed to Section 10.2.

10.1.3.2.1.7 NOTE: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor.

10.1.3.2.2 Continuous Liquid-Liquid Extraction With Hydrophobic Membrane

10.1.3.2.2.1 Follow the manufacturer's instructions for set-up.

10.1.3.2.2.2 Measure out each 1 L sample aliquot in a separate, clean graduated cylinder. Measure the pH of each sample with wide range pH paper and adjust the pH to between 5 and 9 with 10 N sodium hydroxide or concentrated sulfuric acid, if required. Record the pH of the sample "as received" and the exact volume used for extraction on the sample preparation log and on the Form I PCB. Samples requiring pH adjustment must be noted in the SDG Narrative.

10.1.3.2.2.3 Using a syringe or volumetric pipet, add 1.0 ml of the surrogate standard spiking solution (0.2 µg/ml/Section 7.2.4.1.1) into the sample and mix well.

10.1.3.2.2.4 Rinse the graduated cylinder with 30 ml of methylene chloride and transfer the rinsate to the continuous extractor. Rinse the empty 1 L container with 30 ml of methylene chloride and add the rinsate to the continuous extractor.

10.1.3.2.2.5 Add sufficient methylene chloride to the continuous extractor to ensure proper solvent cycling during operation. Adjust the drip rate to 15 ml/minute (recommended); optimize the extraction drip rate. Extract for a minimum of 6 hours. (NOTE: Due to the smaller volume of solvent used during the extraction process, some sample matrices (e.g., oily samples, samples containing a high concentration of surfactants) may create an emulsion which will consume the solvent volume, preventing the efficient extraction of the sample. When this occurs, add additional solvent to assure efficient extraction of the sample, and extend the extraction time for a minimum of 6 hours. If the sample matrix prevents the free flow of solvent through the membrane, then the non-hydrophobic membrane continuous liquid-liquid type extractor must be used.) Allow to cool, then detach the distillation flask. Proceed to Section 10.2.

10.1.3.2.2.6 NOTE: Some continuous liquid-liquid extractors are also capable of concentrating the extract within the extraction set-up. Follow the manufacturer's instructions for concentration when using this type of extractor. Using the hydrophobic membrane, it may not be necessary to dry the extract with sodium sulfate.

10.1.3.2.3 The Contractor may choose to use alternate continuous liquid-liquid extractor types. However, the alternate extractor must be used for all extractions and must meet all the method technical acceptance criteria established in the SOW. When using alternate extractors or design types, follow the manufacturer's instructions for set-up and operation.

10.1.4 Soil/Sediment/Solid Samples

The "sample" is defined as the entire contents of the sample container. Do not discard any supernatant liquids. Just prior to removing the sample for pH determination, percent moisture analysis and/or extraction, mix the contents of the sample container thoroughly either by gentle shaking or with a narrow metal spatula. Remove and discard any large foreign objects such as sticks, leaves, and rocks in soil samples. For other types of solid materials, break the sample into small soil-like pieces with a metal spatula to increase the surface area.

10.1.4.1 pH Determination

Transfer 50 g of soil/sediment/solid to a 100 ml beaker. Add 50 ml of water and continuously stir for 1 hour on a magnetic stirrer. Determine the pH of the sample with a calibrated pH meter while the sample is stirring. Report the pH value on the sample preparation log and on the Form I PCB. If the pH of the soil/sediment/solid is greater than 9 or less than 5, document the

EPA sample number and the exact pH in the SDG Narrative but do not attempt to adjust the pH of the sample. Discard the portion of sample used to determine the pH. If limited sample volume is received use a smaller 1:1 ratio of grams of soil/sediment/solid sample to mls of reagent water for the pH determination. NOTE: The minimum grams to water ratio for pH determination shall be 5 g to 5 ml. The Contractor must note any deviations to the method in the SDG Narrative.

10.1.4.2 Percent Moisture

10.1.4.2.1 Prior to sample extraction and analysis, determine the sample's percent moisture. Weigh 5-10 g of the soil/sediment/solid into a tared crucible and dry overnight or for at least 12 hours in an oven at 105 °C. Allow the sample to cool in a desiccator before reweighing. Calculate the percent moisture using the equation below. Aroclor concentrations will be reported relative to the dry weight of soil/sediment/solid.

EQ. 6

$$\%\ Moisture = \frac{grams\ of\ wet\ sample - grams\ of\ dry\ sample}{grams\ of\ wet\ sample} \times 100$$

10.1.4.2.2 If the percent moisture of the sample as determined above is less than 70 percent (< 70 %) , proceed with extraction and analysis for soil/sediment/solid samples as described in Section 10.1.4.3.

10.1.4.2.3 If the percent moisture of the soil/sediment/solid sample as determined above is greater than or equal to 70 percent (≥ 70 %); centrifuge and decant the sample to remove the majority of the water or the sample may be pressure filtered. Determine the percent moisture of the remaining centrifuged/filtered solid sample following Section 10.1.4.2.1 above. If the percent moisture of the centrifuged or filtered soil/sediment/solid sample is less than 70 percent (< 70 %), proceed with extraction and analysis of the centrifuged/filtered sample using the soil/ sediment/solid sample method described in Section 10.1.4.3.

10.1.4.2.4 If the percent moisture of the centrifuged/filtered soil/ sediment/solid sample is greater than or equal to 70 percent (≥70 %), then the Contractor shall contact the RSCC for directions. The Region may require that the Contractor do one of the following:

- Analyze the centrifuged/filtered soil/sediment/solid sample "as is";
- Use an additional aliquot (weight) of centrifuged soil/ sediment/solid sample (≥ 70 %M) for extraction and/or decrease the final extract volume (to no lower than 0.5 ml) to achieve the dry weight CRQLs;
- Use another method of analysis;
- Do not analyze that sample.

10.1.4.2.5 If percent moisture of the centrifuged/filtered soil/sediment/ solid sample is greater than 90 percent (> 90 %), then the Contractor must contact the RSCC for directions. The Region may require that the Contractor do one of the following:

- Analyze the soil/sediment/solid sample (≥ 90 %M) "as is";
- Use an additional aliquot (weight) of soil/sediment/solid sample (≥ 90 %M) for extraction and/or decrease the final extract volume (to no lower than 0.5 ml) to achieve the dry weight CRQLs;

- Use another method of analysis;
- Do not analyze that sample.

10.1.4.2.6 If a sufficient sample weight/volume has not been provided by the sampler to perform the additional percent moisture determinations and/or to extract/analyze an increased portion of sample, then the Contractor shall contact the RSCC to ascertain whether or not the sample should be analyzed.

10.1.4.2.7 For all samples that do not meet the greater than or equal to 70 percent moisture (≥ 70 %M) requirement, the Contractor shall note the problem, the EPA sample numbers for the affected samples, the initial and subsequent percent moisture(s), and the steps taken to achieve the dry weight CRQLs including the sample weight/ volume prepared and analyzed, the final extract volume and the Region's instructions in the SDG Narrative.

10.1.4.3 Soil/Sediment/Solid Extraction by Sonication

10.1.4.3.1 Tune the sonication according to the manufacturer's directions prior to extracting samples by this procedure.

10.1.4.3.2 Weigh approximately 30 g of sample to the nearest 0.1 g into a 400 ml beaker and add 60 g of anhydrous powdered or granulated sodium sulfate. Record the exact weight of sample taken on the sample preparation log and the Form I PCB. The sample and the added sodium sulfate should be a homogeneous, granular mixture at this point.

10.1.4.3.3 Add 1.0 ml of the surrogate standard spiking solution (0.2 µg/ml/ Section 7.2.4.1.1) to all soil/sediment/solid samples by using a volumetric pipet or a syringe. Immediately add 100 ml of 1:1 methylene chloride-acetone to the sample.

10.1.4.3.4 Place the bottom surface of the sonication probe about ½ inch below the surface of the solvent but above the sample layer.

10.1.4.3.5 Sonicate for 3 minutes using a 3/4 inch disruptor horn at full power (output control knob at 10) with pulse on and percent duty cycle knob set at 50 percent. Do <u>not</u> use a microtip. NOTE: These settings refer to the Model W-385. When using a sonication other then Model W-385, refer to the instructions provided by the manufacturer for appropriate output settings.

10.1.4.3.6 The sample extracts can be gravity or vacuum filtered.

10.1.4.3.7 For gravity filtration prepare a filtration/drying bed by placing a plug of glass wool in the neck of a 10 cm powder funnel and filling the funnel to approximately half its depth (4 or 5 cm) with anhydrous sodium sulfate (80-100 g). Decant the extract through the packed funnel and collect it in a 500 ml evaporative (K-D) flask attached to a concentrator tube.

10.1.4.3.8 For vacuum filtration, use Whatman No. 41 paper in the Buchner funnel. Pre-wet the paper with methylene chloride/acetone before decanting the solvent.

10.1.4.3.9 Repeat the extraction two more times with additional 100 ml portions of the 1:1 methylene chloride-acetone. Before each extraction, thoroughly mix the solid residue and make certain that the sodium sulfate is free flowing and not a consolidated mass. As required, break up large lumps with a clean spatula or very carefully, with the tip of the unenergized probe. Decant and filter the extraction solvent after each sonication and combine all three extracts. On the final sonication, pour the entire sample into the funnel and rinse the beaker and funnel with 60 ml of 1:1 methylene chloride/acetone. Proceed to Section 10.2.

10.1.5 Oil and Oily Sludges (Waste)

10.1.5.1 Transfer approximately 1 g (to the nearest 0.1 g) of sample to a

to a 20 ml vial which has been precalibrated to 10 ml. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken on the sample preparation log and the Form I PCB. Cap the vial before proceeding with the next sample to avoid any cross-contamination.

10.1.5.2 Add 2.0 g of anhydrous powdered or granulated sodium sulfate to the sample in the vial and mix well. More sodium sulfate may be added to make sure the sample is free-flowing.

10.1.5.3 Surrogates are added to all samples, QC samples and blanks. Add 0.2 ml of the surrogate standard spiking solution (1.0 µg/ml/Section 7.2.4.1.2) to the sample mixture.

10.1.5.4 Immediately dilute to 10.0 ml with methylene chloride.

10.1.5.5 Cap and sonicate in a sonication water bath for 2 minutes.

10.1.5.6 Proceed to Sections 10.2.2 and 10.2.3 for extract concentration and solvent exchange to hexane.

10.2 Concentrating the Extract

10.2.1 Concentration by K-D

10.2.1.1 Assemble a Kuderna-Danish (K-D) apparatus by attaching a 10 mL concentrator tube to a 500 mL evaporative flask. Other concentration devices or techniques may be used in place of the K-D. If other concentration devices or techniques are used, samples processed using these devices or techniques must meet all the sample technical acceptance criteria established by the SOW.

10.2.1.2 For water sample extracts prepared as described in Section 10.1.3, pour the extract through a drying column containing about 10 cm of anhydrous granular sodium sulfate and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and the column with at least two additional 20 to 30 mL portions of methylene chloride to complete the quantitative transfer.

10.2.1.3 Soil/sediment/solid sample extracts prepared by the procedures described in Section 10.1.4 will result in extracts containing a mixture of acetone and methylene chloride. Transfer the extract directly to the K-D concentrator. Rinse the Erlenmeyer flask with 20-30 mL of methylene chloride to complete the quantitative transfer. Rinse the extraction vial with 20-30 mL of methylene chloride to complete the quantitative transfer.

10.2.1.4 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL methylene chloride to the top of the column. Place the K-D apparatus in a hot water bath (60 °C to 70 °C recommended) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 15 minutes. At the proper rate of distillation, the balls of the column will chatter actively, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 or 2 mL of methylene chloride.

10.2.2 Final Concentration of Extract

Two different techniques are permitted to concentrate the extract to volume before cleanup or solvent exchange or to the final extract volume before instrumental analysis. They are the Micro Snyder Column and Nitrogen Evaporation Techniques.

10.2.2.1 Micro Snyder Column Technique

Add another one or two clean boiling chips to the concentrator tube and attach a two-ball micro Snyder column. Pre-wet the Snyder column by adding about 0.5 mL of hexane to the top of the column. Place the K-D apparatus in a hot water bath (80 °C to 90 °C recommended) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus from the water bath and allow it to drain for at least 10 minutes while cooling. Remove the Snyder column and rinse its flask and lower joint into the concentrator tube with 0.2 mL of hexane. Adjust the final volume to 1 or 2 mL with hexane.

10.2.2.2 Nitrogen Evaporation Technique (taken from ASTM Method D 3086)

10.2.2.2.1 Place the concentrator tube in a warm water bath (30 °C to 35 °C recommended) and evaporate the solvent volume to the final volume by blowing a gentle stream of clean, dry nitrogen

(filtered through a column of activated carbon) onto the solvent. DO NOT ALLOW THE EXTRACT TO GO TO DRYNESS. Adjust the final extract volume to 10.0 mL with hexane. Proceed to Section 10.3 for cleanup procedures.

10.2.2.2.2 Gas lines from the gas source to the evaporation apparatus must be stainless steel, copper, or Teflon tubing. Plastic tubing must not be used between the carbon trap and the sample as it may introduce interferences. The internal walls of new tubing must be rinsed several times with hexane and then dried prior to use.

10.2.2.2.3 During evaporation, the tube solvent level must be kept below the water level of the bath.

10.2.2.3 Proceed with the solvent exchange to hexane described in Section 10.2.3.

10.2.3 Solvent Exchange into Hexane

This procedure applies to water, soil/sediment/solid and/or oil or oily sludge sample extracts.

10.2.3.1 With the extract in a K-D apparatus, remove the Snyder column, add 50 mL of hexane and a new boiling chip, and re-attach the Snyder column. Pre-wet the column by adding about 1 mL of hexane to the top. Concentrate the solvent extract as described previously (Section 10.1.6.1), but increase the temperature of the water bath (to between 80 and 90 °C recommended). When the apparent volume of liquid reaches 3 to 5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. DO NOT ALLOW THE EVAPORATOR TO GO DRY.

10.2.3.2 Remove the Snyder column; rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. Complete the quantitative transfer of the extract to a 10 mL vial by rinsing with hexane.

10.2.3.3 Adjust the final volume of the hexane extracts to 10.0 mL. Proceed to Section 10.3 for cleanup procedures.

10.3 Cleanup Procedures

There are two cleanup procedures specified in this method: sulfuric acid cleanup and sulfur cleanup. Sulfuric acid cleanup must be performed for all water, soil/sediment/solid and oil or oily sludge sample extracts. Sulfur cleanup must be performed for all sample extracts contaminated with sulfur. Blanks and QC samples must be subjected to the same cleanup procedures as the unspiked samples.

10.3.1 Sulfuric Acid Cleanup

10.3.1.1 Introduction to Sulfuric Acid Cleanup

Organic compounds can interfere in the identification and quantitation of PCBs. Thus, a portion of the extract is subjected to sulfuric acid cleanup to improve the identification and quantitation of PCBs.

10.3.1.2 Frequency of Sulfuric Acid Cleanup

Sulfuric acid cleanup must be performed at least once for each water extract, soil/sediment/solid extract, oil and oily sludge waste dilutions and all associated blanks and QC samples.

10.3.1.3 Procedure for Sulfuric Acid Cleanup

10.3.1.3.1 Using a syringe or a volumetric pipette, transfer 1.0 mL of the hexane extract to a 10 mL vial and, in a fume hood, carefully add 5 mL of the 1:1 sulfuric acid solution.

CAUTION: Make sure that there is no exothermic reaction nor evolution of gas prior to proceeding.

10.3.1.3.2 Cap the vial tightly and shake gently for one minute. CAUTION: Stop immediately if the vial leaks. Avoid skin contact, sulfuric acid burns.

10.3.1.3.3 Allow the phases to separate for at least 1 minute. Examine the top (hexane) layer; it should not be highly colored nor should it have a visible emulsion or cloudiness.

10.3.1.3.4 If a clean phase separation is achieved, carefully remove the hexane layer and transfer to a clean vial. Proceed to Section 10.3.1.3.6.

10.3.1.3.5 If the hexane layer is colored or the emulsion persists for several minutes, remove the sulfuric acid layer (dispose properly) and repeat the cleanup procedure with additional 5 ml aliquots of clean 1:1 sulfuric acid until the hexane extract is no longer colored. Allow the phases to separate and transfer the hexane layer to a clean vial.

10.3.1.3.6 Add an additional 1 ml of hexane to the sulfuric acid layer, cap and shake. This second extraction is done to ensure quantitative transfer of the PCBs.

10.3.1.3.7 Remove the second hexane layer and combine with the original hexane layer.

10.3.1.3.8 Return the combined hexane extracts to a 1.0 ml final sample volume using either of the final concentration techniques described in section 10.2.2.

10.3.2 Sulfur Cleanup

10.3.2.1 Introduction to Sulfur Cleanup

10.3.2.1.1 Sulfur contamination will cause a rise in the baseline of a chromatogram and may interfere with the analyses of the later eluting PCBs. If crystals of sulfur are evident or if the presence of sulfur is suspected, sulfur removal must be performed. Interference due to sulfur is not acceptable. Sulfur can be removed by one of the two techniques, detailed below. The Contractor must specify which technique was used on the sample preparation log and in the SDG narrative. If the sulfur concentration is such that crystallization occurs in the concentrated extract, centrifuge the extract to settle the crystals, and remove the sample extract with a disposable pipette, leaving the excess sulfur in the centrifuge tube. Transfer the extract to a clean centrifuge tube or clean concentrator tube before proceeding with further sulfur cleanup.

10.3.2.1.2 If only part of a set of samples require sulfur cleanup, then a sulfur cleanup blank is required for that part of the set (Section 12.1.4).

10.3.2.2 Frequency of Sulfur Cleanup

Sulfur removal is required for all sample extracts that contain sulfur.

10.3.2.3 Procedure for Sulfur Cleanup

10.3.2.3.1 Mercury Technique

Add one to three drops of mercury to each 1.0 ml aliquot of the hexane extract in a clean vial. Tighten the top on the vial and agitate the sample for 30 seconds. Filter or centrifuge the extract. Pipet the hexane extract to another vial and leave all solid precipitate and liquid mercury. If the mercury appears shiny, proceed to Section 10.4 for GC/ECD analysis. If the mercury turns black, repeat sulfur removal as necessary. The extract transferred to the vial still represents the 1.0 ml final volume. CAUTION: Waste containing mercury should be segregated and disposed of properly. NOTE: Mercury is highly

toxic and therefore must be used with great care. Prior to using mercury, it is recommended that the analyst become acquainted with proper handling and cleanup techniques associated with mercury.

10.3.2.3.2 Copper Technique

Add approximately 2 g of cleaned copper powder to a 1.0 ml aliquot of the hexane extract in a centrifuge or concentrator tube (2 g will fill the tube to about the 0.5 ml mark). Mix the copper and extract for at least 1 minute on a mechanical shaker. Separate the hexane extract from the copper powder by drawing off the extract with a disposable pipet, and transfer to a clean GC vial. The extract transferred to the vial still represents the 1.0 ml final volume. The separation of the extract from the copper powder is necessary to prevent degradation of the sample. If the copper appears bright, proceed to Section 10.4 for GC/ECD analysis. If the copper changes color, repeat the sulfur removal procedure as necessary.

10.4 GC/ECD Analysis

10.4.1 Introduction to Sample Analysis by GC/ECD

10.4.1.1 Before samples, QC samples or required blanks can be analyzed, the instrument must meet the initial calibration and continuing calibration technical acceptance criteria. Sample analysis on both GC columns is required for all samples, blanks and QC samples.

10.4.1.2 Sample extracts, standards, QC samples and blanks must be analyzed within an analytical sequence as defined in Section 10.4.2.1, under the same instrumental conditions.

10.4.1.3 Set up the GC/ECD system per the requirements in Section 9.0. Unless ambient temperature on-column injection is used (see Section 9.1.4), the injector must be heated to at least 200 °C. The optimized gas chromatographic conditions established in Section 9.1 must be used for all analyses.

10.4.2 Procedure for Sample Analysis by GC/ECD

The injection must be made on-column by using either automatic or manual injection. If autosamplers are used, 1 µL injection volumes may be used. Manual injections must use at least 2 µL injection volumes. The same injection volume must be used for all standards, samples, QC samples, and blanks associated with the same initial calibration. If a single injection is used for two GC columns attached to a single injection port, it may be necessary to increase the injection volume. However, the same injection volume must be used for all analyses.

10.4.2.1 Analytical Sequence

The initial calibration analytical sequence is presented in Section 9.2.3.4.1. At least one three point calibration curve must be analyzed with each analytical sequence in order to demonstrate instrument linearity. At a minimum, the calibration will include a three point calibration curve of the target Aroclor (or Aroclor 1254, if unknown) to demonstrate the linearity of the GC/ECD as well as analysis of one concentration level of each of the other individual Aroclor standards. NOTE: The final two steps of the initial calibration sequence, with the injection of the instrument blank and the continuing calibration standard, mark the beginning of the first 12-hour analytical sequence in which samples may be run. All samples must be analyzed within a valid analytical sequence as given below.

Time	Injection #	Material Injected
	1 - 10	Initial calibration sequence (Section 9.2.3.4.1)

Time	Injection #	Material Injected
0 hr.	11	Instrument Blank
	12	Midpoint Aroclor Continuing calibration standard
	13	Additional continuing calibration standards or Samples
	etc...	Last sample
12 hr.	1st injection past 12:00 hr.	Instrument blank
	2nd injection	Midpoint Aroclor Continuing calibration standard
	etc...	Additional continuing calibration standards and/or samples
24 hr.	0	Last sample
	1st injection past 12 hr.	Instrument blank
	2nd injection past 12 hr.	Midpoint Aroclor Continuing calibration standard
	etc...	Additional continuing calibration standards and/or samples
36 hr.	0	Last sample
	1st injection past 12 hr.	Instrument blank
	2nd injection	Midpoint Aroclor Calibration verification standard(s)

10.4.2.1.1 The first 12 hours are counted from the injection of the instrument blank at the end of the initial calibration sequence, not from injection #1. Samples may be injected until 12 hours have elapsed. All subsequent 12-hour periods are timed from the injection of the instrument blank that brackets the front end of the samples. Because the 12-hour time period is timed from injection of the instrument blank until the injection of the last sample, each 12-hour period may be separated by the length of one chromatographic run, that of the analysis of the last sample. While the 12-hour period may not be exceeded, the laboratory may run instrument blanks and continuing calibration standards more frequently, for instance, to accommodate staff working 8-hour shifts.

10.4.2.1.2 After the initial calibration, the analytical sequence may continue as long as acceptable instrument blanks, and midpoint continuing calibration standards are analyzed at the required frequency. This analytical sequence shows only the minimum required blanks and standards. More blanks and standards may be run at the discretion of the Contractor; these must also satisfy the criteria presented in Section 9 in order to continue the analytical sequence.

10.4.2.1.3 An analytical sequence must also include all required QC samples and method blank analyses (including sulfuric acid and/or sulfur blanks), but the Contractor may decide at what point in the sequence they are to be analyzed.

10.4.2.1.4 The requirements for the analytical sequence apply to both GC columns and for all instruments used for these analyses.

10.4.3 Sample Dilutions

10.4.3.1 All samples must be analyzed at the most concentrated level that is consistent with achieving satisfactory chromatography (defined in Section 11.3).

10.4.3.2 If the response of the largest peak of a single Aroclor component in any sample extract is greater than the response of that single Aroclor component in the initial calibration high point standard, then the sample extract must be diluted to have the response of the largest peak in the detected Aroclor between the responses of the initial calibration midpoint and high point standards of that Aroclor peak. The chromatographic data from the diluted analysis of any Aroclor must be reported at greater than 25.0 percent but less than 100.0 percent of full scale.

10.4.3.3 If dilution is employed solely to get a multicomponent pattern on scale, then the Contractor must report data for both analyses.

10.4.3.4 If the Contractor has determined from site history or sample extract screening that dilution prior to sample analysis will be necessary, an undiluted sample analysis may not be required. If an acceptable chromatogram (as defined in Section 11.3) is achieved with the diluted extract, an additional extract 10 times more concentrated than the diluted sample must be also injected and reported with the sample data.

10.4.3.5 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram. If the chromatogram of any sample needs to be replotted electronically to meet these requirements, then both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

10.4.5.6 Do not submit data for more than two analyses, i.e., the original sample extract and one dilution, or, if a screening procedure was employed, from the most concentrated dilution analyzed and one further dilution. This statement does not refer to reanalyses required due to failed technical acceptance criteria.

10.4.5.7 The Contractor may receive instructions with the sampling paperwork which prohibits sample dilutions under any circumstances. This may be required in instances where the CRQLs for most target compounds must be achieved even though one or more target compounds exceed the calibration range. In these cases, if screening results indicate that sample dilution is required, then the Contractor shall contact the RSCC to ascertain whether or not that sample should be analyzed at a dilution. For all samples requiring dilutions, the Contractor must note the problem, the EPA sample numbers and any Regional instructions in the SDG Narrative.

10.4.6 Procedure for Non-Anticipated Sample Results

If an Aroclors is identified in a sample during the analytical sequence (Section 9.2.3.4), the Contractor must perform a three point calibration curve for that Aroclor within 72 hours of the first detection in any sample. The steps must be followed to assure that the identified Aroclor is correctly identified and quantitated.

10.4.6.1 If the Aroclor detected was the same as that used for the initial three point calibration (Section 9.2.3) and the samples were analyzed during a valid 12-hour analytical sequence, proceed with quantitation as described in Section 11.0.

10.4.6.2 If additional Aroclors are identified during the analytical sequence (Section 9.2.3), the Contractor must perform a three point calibration curve for that Aroclor within 72 hours of the first detection in any sample and within a valid 12-hour analytical sequence.

- The three point calibration curve must meet all initial calibration technical acceptance criteria and the midpoint Aroclor standard, which was analyzed during the initial calibration sequence must meet the continuing calibration technical acceptance criteria before any sample results can be reported. Proceed with quantitation of the sample results as described in Section 11.2.

- If the initial calibration technical acceptance criteria are

not met, follow the appropriate corrective actions in Section 9.2.6. Reanalyze the initial calibration for that Aroclor and reanalyze the affected samples within a valid 12-hour analytical sequence (using the detected Aroclor as the continuing calibration standard).

! If the initial calibration technical acceptance criteria are met, but the continuing calibration technical acceptance criteria are unacceptable; then prepare a fresh continuing calibration standard (Section 7.2.4.4). Analyze an instrument blank and the fresh continuing calibration standard. If blank and continuing calibration technical acceptance criteria are met for the reanalyzed blank/standard pair, then reanalyze the associated samples within a valid 12-hour analytical sequence (using the detected Aroclor as the final continuing calibration standard).

11.0 DATA ANALYSIS AND CALCULATIONS

11.1 Qualitative Identification

11.1.1 Identification of Target Compounds

11.1.1.1 A set of three to five major peaks is selected for each Aroclor. The retention time window for each peak is determined from the initial calibration analysis. Identification of any Aroclor in a sample is based on pattern recognition in conjunction with the elution of three to five sample peaks within the retention time windows of the corresponding peaks of the standard on both GC columns. Aroclors are quantitated using the mean calibration factor of the three point initial calibration. The number of potential quantitation peaks for each Aroclor are listed in Table 2.

11.1.1.2 The choice of the peaks used for Aroclor identification and the recognition of those peaks may be complicated by the environmental alteration of the Aroclors, and by the presence of co-eluting analytes or matrix interferences, or both. Because of the alteration of these materials in the environment, multicomponent analytes in samples may give patterns similar to, but not identical with, those of the standards.

11.1.1.3 If more than one Aroclor is observed in a sample, the Contractor must choose different peaks to quantitate each Aroclor. A peak common to both Aroclors present in the sample must not be used to quantitate either compound.

11.1.2 GC/MS Confirmation of Aroclors

11.1.2.1 Any Aroclor listed in Exhibit C for which a concentration is reported from a GC/ECD analysis must have the identification confirmed by GC/MS, if the concentration is sufficient for that purpose. The following guidance should be used in performing GC/MS confirmation. If the Contractor fails to perform GC/MS confirmation as appropriate, the Agency may require reanalysis of any affected samples at no additional cost to the Agency.

11.1.2.2 The GC/MS confirmation may be accomplished by one of three general means:

- Examination of the semivolatile GC/MS library search results (i.e., TIC data), or
- A second analysis of the semivolatile extract, or
- Analysis of the Aroclor extract, following any solvent exchange and concentration steps that may be necessary.

11.1.2.3 The semivolatile GC/MS analysis procedures outlined in Exhibit D - SVOA are based on the injection into the instrument of approximately 20 ng of a target compound in a 2 µL volume. The semivolatile CRQL values in Exhibit C are based on the sample concentration that corresponds to an extract concentration of 10 ng/µL of target analyte. However, these are quantitation limits, and the detection of analytes and generation of reproducible mass spectra will routinely be possible at levels 3-10 times lower. The sample concentration corresponding to 10 ng/µL in extract will depend on the sample matrix.

11.1.2.3.1 For water samples, 20 ng/2 µL corresponds to a sample concentration of 10 µg/L.

11.1.2.3.2 For soil/sediment/solid samples prepared according to the semivolatile low level soil/sediment/solid method (i.e., 30 g of soil/sediment/solid), the corresponding sample concentration is 330 µg/Kg.

11.1.2.3.3 Therefore, based on the values given above, any sample in which Aroclor concentration in the sample extract is greater than or equal to 50 ng/μL should enable the laboratory to confirm the

Aroclor by GC/MS analysis of the semivolatile extract.

11.1.2.4 In order to confirm the identification of the target Aroclor, the laboratory must also analyze a reference standard for the analyte. In order to demonstrate the ability of the GC/MS system to identify the analyte in question, the concentration of the standard should be 50 ng/μL for all Aroclors, except 100 ng/μL for Aroclor 1221.

11.1.2.5 The laboratory is advised that library search results from the NIST/EPA/NIH (May 1992 release or most recent release) and Wiley (1991 release or most recent release) mass spectral library will not likely list the name of the Aroclor analyte as it appears in this SOW, hence, the mass spectral interpretation specialist is advised to compare the CAS Registry numbers for the individual polychlorinated biphenyls (PCBs) to those from the library search routine.

11.1.2.6 If the analyte cannot be confirmed from the semivolatile library search data for the original semivolatile GC/MS analysis, the laboratory may analyze another aliquot of the semivolatile sample extract after further concentration of the aliquot. This second aliquot must either be analyzed as part of a routine semivolatile GC/MS analysis, including instrument performance checks (DFTPP), calibration standards containing the Aroclors as described in Section 11.1.2.4, or it must be analyzed along with separate reference standards for the analytes to be confirmed.

11.1.2.7 If the analyte cannot be confirmed by either of the procedures above, then an aliquot of the extract prepared for the GC/ECD analysis must be analyzed by GC/MS, following any necessary solvent exchange and concentration steps. As in Section 11.1.2.4, analysis of a reference standard is required.

11.1.2.8 Regardless of which of the three approaches above is used for GC/MS confirmation, the appropriate blank must also be analyzed by GC/MS to demonstrate that the presence of the analyte was not the result of laboratory contamination. If the confirmation is based on the analysis of the semivolatile extract, then the semivolatile method blank extracted with the sample must also be analyzed. If the confirmation is based on the analysis of the extract prepared for the GC/ECD analysis, then the method blank extracted with the samples must also be analyzed.

11.1.2.9 If the identification of the analyte cannot be confirmed by any of the GC/MS procedures above and the concentration calculated from the GC/ECD analysis is greater than or equal to the concentration of the reference standard analyzed by GC/MS, then report the analyte as undetected, adjust the sample quantitation limit (the value associated with the "U" qualifier) to a sample concentration equivalent to the concentration of the GC/MS reference standard, and qualify the results on Form I with one of the laboratory-defined qualifiers ("X," "Y," or "Z"). In this instance, define the qualifier explicitly in the SDG Narrative, and describe the steps taken to confirm the analyte in the SDG Narrative.

11.1.2.10 For GC/MS confirmation of Aroclors, the required deliverables are copies of the library search results (best TIC matches) and the spectra of the three characteristic peaks for both the sample and the reference standard.

11.1.2.11 The purpose of the GC/MS analysis for the Aroclors is to confirm the presence of the polychlorinated biphenyls. The GC/MS analytical results for the Aroclors shall not be used for quantitation and the GC/MS results shall not be reported. The exception noted in Section 11.1.2.9 applies only to analytes that cannot be confirmed above the reference standard concentration.

11.2 Calculations

11.2.1 Target Compounds

The quantitation of Aroclors must be accomplished by comparing the

heights or the areas of each of the three to five major peaks of the Aroclor in the sample with the mean calibration factor for the same peaks established during the initial calibration sequence. The concentration of multicomponent analytes is calculated by using Equations 7 and 8, where A_x is the area for each of the major peaks of the multicomponent analyte. The concentration of each peak is determined and then a mean concentration for the three to five major peaks is determined on each column. The concentrations of the surrogates are calculated separately for both GC columns using the same equations.

11.2.1.1 Water

EQ. 7

$$Concentration\ \mu g/L = \frac{(A_x)(V_t)(Df)}{(\overline{CF})(V_o)(V_i)}$$

Where;

$\overline{CF}$ = Mean Calibration factor from the initial calibration.

A_x = Area or height of the peak for the compound to be measured.

V_o = Volume of water extracted in milliliters (ml).

V_i = Volume of extract injected in microliters (µL). (If a single injection is made onto two columns, use one half the volume in the syringe as the volume injected onto each column.)

V_t = Volume of the concentrated extract in microliters (µL). (V_t = 10,000 µL)

Df = Dilution factor. The dilution factor for analysis of water samples by this method is defined as follows:

$$\frac{\text{µL most concentrated extract + µL clean solvent}}{\text{µL most concentrated extract}}$$

If no dilution is performed, Df = 1.0.

11.2.1.2 Soil/Sediment/Solid and Oily Sludge (waste)

EQ. 8

$$Concentration\ \mu g/Kg\ (Dry\ weight\ basis) = \frac{(A_x)(V_t)(Df)}{(\overline{CF})(V_i)(W_s)(D)}$$

Where,

A_x, V_t, V_i, Df and CF are as given for water, above.

$D = \frac{100 - \%\ moisture}{100}$ (soil/sediment/solid only)

W_s = Weight of sample extracted in grams (g).

11.2.1.3 Because of the likelihood that compounds co-eluting with the target compounds will cause positive interferences and increase the concentration determined by the method, the lower of the two concentrations calculated for each Aroclor is reported on Form I PCB. In addition, the concentrations calculated for both the GC columns are reported on Form XI, along with a percent difference (%D) comparing the two concentrations. The percent difference is calculated according to Equation 9.

EQ. 9

$$\%D = \frac{Conc_H - Conc_L}{Conc_L} \times 100$$

Where,

$Conc_H$ = The higher of the two concentrations for the Aroclor in question

$Conc_L$ = The lower of the two concentrations for the Aroclor in question

11.2.1.4 Note that using this equation will result in percent difference values that are always positive. The value will also be greater than a value calculated using the higher concentration in the denominator; however, given the likelihood of a positive interference raising the concentration determined on one GC column, this is a conservative approach to comparing the two concentrations.

11.2.2 CRQL Calculation

Sample specific CRQLs must be calculated and reported on Form I PCB. If the adjusted CRQL is less than the CRQL listed in Exhibit C PCBs, report the CRQL in Exhibit C.

11.2.2.1 Water Samples

EQ. 10

$$\underset{CRQL}{Adjusted} = \underset{CRQL}{Contract} \times \frac{(V_x)(V_t)(V_y)(Df)}{(V_o)(V_c)(V_i)}$$

Where,

V_t, Df, V_o, and V_i are as given in equation 7.
V_x = Contract sample volume (1000 ml).
V_y = Contract injection volume (1 µL or 2 µL).
V_c = Contract concentrated extract volume (10,000 µL).

11.2.2.2 Soil/Sediment/Solid or Oil/Oily Sludge (waste) Samples

EQ. 11

$$\underset{CRQL}{Adjusted} = \underset{CRQL}{Contract} \times \frac{(W_x)(V_t)(V_y)(Df)}{(W_s)(V_c)(V_i)(D)}$$

Where,

V_t, Df, W_s, V_i and D are as given in equation 8.
W_x = Contract sample weight (30 g soil/sed/solid or 1 g for oil and oily sludge).
V_y = Contract injection volume (1 µL or 2 µL).
V_c = Contract concentrated extract volume (10,000 µL).

11.2.3 Surrogate Recoveries

11.2.3.1 The concentrations of the surrogates are calculated separately for each GC column in a similar manner as the other analytes, using Equations 7 and 8. Use the mean calibration factor from the initial calibration curve. The recoveries of the surrogates are calculated for each GC column according to Equation 11, below.

EQ. 12

$$Percent\ Recovery = \frac{Q_d}{Q_a} \times 100$$

Where,

Q_d = Quantity determined by analysis
Q_a = Quantity added

11.2.3.2 The advisory limits for the recovery of the surrogates are 30 - 150 percent for both surrogate compounds.

11.2.3.3 As these limits are only advisory, no further action is required by the laboratory; however, frequent failures to meet the recovery limits for surrogates warrant investigation by the laboratory, and may result in questioning by the Agency. Surrogate recovery data from both GC columns are reported on Form II PCB (see Exhibit B).

11.3 Technical Acceptance Criteria for Sample Analysis

The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on each GC column.

11.3.1 Samples must be analyzed under the GC/ECD operating conditions in Section 9.0. The instrument must have met all initial calibration, continuing calibration and blank technical acceptance criteria. Samples must be cleaned-up with sulfuric acid and also for sulfur removal, when required. Sample data must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks, and midpoint Aroclor continuing calibration standards, as described in Section 10.4.2.1.

11.3.2 The samples must be extracted and analyzed or reextracted and reanalyzed within the contract required holding times defined in Section 8.4.

11.3.3 The samples must have an associated method blank(s) meeting the technical acceptance criteria for method blanks. All samples must have an associated sulfuric acid cleanup blank meeting the technical acceptance criteria for sulfuric acid cleanup blanks. When sulfur cleanup blanks are required, the samples must have associated with them a sulfur cleanup blank meeting the technical acceptance criteria for sulfur cleanup blanks.

11.3.4 The retention time for each of the surrogates must be within the retention time windows as calculated in Section 9.2.4.2 for both GC columns.

11.3.5 No target analyte concentrations may exceed the upper limit of the initial calibration range, or else the extract must be diluted and reanalyzed as described in Section 10.4.3.

11.3.6 A three point calibration curve for any identified Aroclor must be analyzed during a valid 12-hour analytical sequence on the same instrument and column, within 72 hours of its detection in a sample.

11.3.7 The identification of Aroclors is based primarily on pattern recognition and on peak retention times displayed on a chromatogram. Therefore, the following requirements apply to all data presented for Aroclor quantitation.

11.3.7.1 When no analytes are identified in a sample, the chromatograms from the analyses of the sample extract must use the same scaling factor as was used for the low point standard of the initial calibration standard associated with those analyses.

11.3.7.2 Chromatograms must display the largest peak of any Aroclor detected in the sample at less than full scale.

11.3.7.3 If an extract must be diluted, chromatograms of any diluted sample extract must display the peaks chosen for quantitation between 25 and 100 percent of full scale.

11.3.7.4 If a chromatogram is replotted electronically to meet these requirements, the scaling factor used must be displayed on the chromatogram.

11.3.7.5 If the chromatogram of any sample needs to be replotted electronically to meet these requirements, both the initial chromatogram and the replotted chromatogram must be submitted in the data package.

11.4 Corrective Action for Sample Analysis

11.4.1 Sample analysis technical acceptance criteria MUST be met before data are reported. Samples contaminated from laboratory sources or associated with a contaminated method blank, sulfuric acid cleanup blank or sulfur cleanup blank will require re-extraction and reanalysis at no additional cost to the Agency. Any samples analyzed that do not meet the technical acceptance criteria will require re-

extraction and/or reanalysis at no additional cost to the Agency. Reextraction and/or reanalysis must be completed within the contract required holding times and must meet all technical acceptance criteria.

11.4.2 If the sample analysis technical acceptance criteria are not met, check calculations, surrogate solutions, and instrument performance. It may be necessary to recalibrate the instrument or take other corrective action procedures to meet the technical acceptance criteria, in which case, the affected samples must be reextracted or reanalyzed at no additional cost to the Agency after the corrective action.

11.4.3 If sample chromatograms have a high baseline or interfering peaks, inspect the system to determine the cause of the problem (e.g. carryover, column bleed, dirty ECD, contaminated gasses, leaking septum, etc.). After correcting the problem, analyze an instrument blank to demonstrate that the system is functioning properly. Reanalyze the sample extracts. If the problem with the samples still exists, then those samples must be re-extracted and reanalyzed. Samples which do not meet the technical acceptance criteria after one re-extraction and the two-step cleanup (sulfuric acid and sulfur cleanups) are reported in the SDG Narrative by EPA Sample Number with a summary of the problem and do not require further analysis.

11.4.4 If the technical acceptance criteria for initial calibration, continuing calibration and/or method blanks are not met, then the contractor must stop and correct the problem before continuing the analytical sequence. Any samples analyzed when the above technical acceptance criteria have not been met must be reanalyzed at no additional cost to the agency. Reanalysis must be completed within the contract required holding times and must meet all technical acceptance criteria.

11.4.5 Sample analyses reported with non-compliant initial calibration, continuing calibration or required blanks shall be subject to a commensurate reduction in sample price or zero payment due to data rejection, depending upon the impact of the non-compliance on data usability.

12.0 QUALITY CONTROL

12.1 Blank Analyses

12.1.1 Introduction

There are two types of blanks always required by this method: the method blank and the instrument blank. Since all the samples submitted for PCB analysis will be subjected to sulfuric acid cleanup, then the method blank must also be subjected to sulfuric acid cleanup, and no separate sulfuric acid method blank will be required. A separate sulfur cleanup blank is also required for all samples subjected to sulfur cleanup. The method and sulfur cleanup blanks must meet their respective technical acceptance criteria.

12.1.2 Method Blanks

12.1.2.1 Summary of Method Blanks

A method blank is a volume of a clean reference matrix (reagent water for water samples, or purified sodium sulfate for soil/sediment/solid samples and/or oil and oily sludge (waste) samples) that is carried through the entire extraction and analytical procedure, including sulfuric acid cleanup. The volume or weight of the reference matrix must be approximately equal to the volume or weight of samples associated with the blank. The purpose of a method blank is to determine the levels of contamination associated with the processing and analysis of samples.

12.1.2.2 Frequency of Method Blanks

A method blank must be extracted once for the following, whichever is most frequent, and analyzed on each GC/ECD system used to analyze samples:

- Each SDG (not to exceed 20 field samples), or
- Each matrix within an SDG, or
- Whenever samples are extracted and/or cleaned-up by the same procedure (separatory funnel, continuous liquid-liquid extraction, sonication and/or waste dilution).

12.1.2.3 Procedure for Method Blank Preparation

12.1.2.3.1 For PCB analyses, a method blank for water samples consists of a 1 L volume of reagent water spiked with 1.0 ml of the surrogate standard spiking solution (Section 7.2.4.1.1). For soil/ sediment/solid samples, the method blank consists of 30 g of sodium sulfate spiked with 1.0 ml of the surrogate standard spiking solution (Section 7.2.4.1.1). For oil or oily sludge (waste) samples, a method blank consists of 9.8 ml of hexane spiked with 0.2 ml of the oily sludge surrogate standard spiking solution (Section 7.2.4.1.2).

12.1.2.3.2 Extract, concentrate, cleanup, analyze and report method blanks following the procedures in Sections 10.0 and 11.0.

12.1.2.4 Technical Acceptance Criteria for Method Blanks

12.1.2.4.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on both GC columns.

12.1.2.4.2 All method blanks must be prepared and analyzed at the frequency described in Section 12.1.2.2 using the procedure above and on a GC/ECD system meeting the initial calibration and continuing calibration technical acceptance criteria. All Aroclor method blanks must undergo sulfuric acid cleanup. Method blanks must be bracketed at 12-hour intervals (or less), in an acceptable analytical sequence, by compliant analyses of instrument blanks and midpoint Aroclor continuing calibration

standards as described in Section 10.4.2.1.

12.1.2.4.3 The concentration of the target compounds (Exhibit C, PCB) in the method blank must be less than the CRQL for each target Aroclor.

12.1.2.4.4 The method blank must meet all sample technical acceptance criteria in Sections 11.3.4 through 11.3.7.

12.1.2.4.5 Surrogate recoveries must fall within the acceptance windows of 30-150%. In the case of the method blank(s), these limits are not advisory.

12.1.2.5 Corrective Action for Method Blanks

12.1.2.5.1 If a method blank does not meet the technical acceptance criteria, the Contractor must consider the system to be out of control.

12.1.2.5.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective action measures must be taken and documented before further sample analysis proceeds. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and sample processing hardware that lead to discrete artifacts and/or elevated baselines be investigated and appropriate corrective actions be taken and documented before further sample analysis. All samples associated with a contaminated method blank must be re-extracted/reanalyzed at no additional cost to the Agency.

12.1.2.5.3 If the surrogate recoveries in the method blank do not meet the acceptance criteria listed in 12.1.2.4.5, first reanalyze the method blank. If surrogate recoveries do not meet the acceptance criteria after reanalysis, the method blank and all samples associated with that method blank must be re-extracted and reanalyzed at no additional cost to the Agency.

12.1.2.5.4 If the method blank failed to meet the technical acceptance criteria listed in Sections 12.1.2.4.2 and 12.1.2.4.4, then there is an instrument problem. Correct the instrument problem and reanalyze the method blank.

12.1.2.5.5 If any technical acceptance criteria (Section 12.1.2.4) for blank analyses are not met, then the contractor must stop and correct the problem before continuing the analytical sequence. If sample analyses are reported with non-compliant blanks, then the contractor may receive a commensurate reduction in sample price or zero payment depending upon the impact of the non-compliance on data usability.

12.1.3 Sulfur Cleanup Blanks

12.1.3.1 Summary of Sulfur Cleanup Blanks

The sulfur cleanup blank is a modified form of the method blank. The sulfur cleanup blank is hexane spiked with the surrogates and carried through the sulfur cleanup and analysis procedures. The purpose of the sulfur cleanup blank is to determine the levels of contamination associated with the separate sulfur cleanup steps.

12.1.3.2 Frequency of Sulfur Cleanup Blanks

The sulfur cleanup blank is prepared separately when only part of a set of samples extracted together requires sulfur removal. A method blank is associated with the entire set of samples. The sulfur cleanup blank is associated with the part of the set which required sulfur cleanup. If all the samples associated with a given method blank are subjected to sulfur cleanup, then the method blank must be subjected to sulfur cleanup, and no separate sulfur cleanup blank is required.

12.1.3.3 Procedure for Sulfur Cleanup Blank

12.1.3.3.1 The concentrated volume of the blank must be the same as the final volume of the samples associated with the blank. The sulfur blank must also contain the surrogates at the same concentrations as the sample extracts (assuming 100.0 percent recovery). Therefore, add 0.1 ml of the surrogate solution to 0.9 ml of hexane in a clean vial, or for a sulfur blank with a final volume of 2 ml, add 0.2 ml of the surrogate solution to 1.8 ml of hexane in a clean vial.

12.1.3.3.2 Proceed with the sulfur removal using the same technique (mercury or copper/Section 10.3.2.3) as the samples associated with the blank.

12.1.3.3.3 Analyze the sulfur cleanup blank according to Section 10.4. Assuming that the material in the sulfur cleanup blank resulted from the extraction of a 1 L water sample, calculate the concentration of each analyte using the equation in Section 11.2.1.1. Compare the results to the CRQL values for water samples in Exhibit C, PCBs.

12.1.3.4 Technical Acceptance Criteria For Sulfur Cleanup Blanks

12.1.3.4.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on both GC columns.

12.1.3.4.2 All sulfur cleanup blanks must be prepared and analyzed at the frequency described in Section 12.1.4.2 using the procedure referenced in Section 12.1.4.3 on a GC/EC system meeting the initial calibration and calibration verification technical acceptance criteria.

12.1.3.4.3 Sulfur cleanup blanks must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks and midpoint Aroclor calibration verification standards as described in Section 10.4.2.1.

12.1.3.4.4 The concentration of the target compounds (Exhibit C, PCBs) in the sulfur cleanup blank must be less than the CRQL for each target compound.

12.1.3.4.5 The sulfur cleanup blank must meet all sample analysis technical acceptance criteria in Sections 11.3.4 to 11.3.7.

12.1.3.4.6 Surrogate recoveries must fall within the recovery limits of 30-150%. In the case of the sulfur cleanup blank, these limits are not advisory.

12.1.3.5 Corrective Action for Sulfur Cleanup Blanks

12.1.3.5.1 If a sulfur cleanup blank does not meet the technical

acceptance criteria, the Contractor must consider the system to be out of control.

12.2.3.5.2 If contamination is a problem, then the source of the contamination must be investigated and appropriate corrective measures must be taken and documented before further sample analysis proceeds. It is the Contractor's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and sample storage and sample processing hardware that lead to discrete artifacts and/or elevated baselines be investigated and appropriate corrective actions be taken and documented before further sample analysis. All samples associated with a contaminated sulfur cleanup blank must be re-extracted/reanalyzed at no additional cost to the Agency.

12.1.3.5.3 If the surrogate recoveries in the sulfur cleanup blank do not meet the acceptance criteria listed in section 12.1.4.4.6, first reanalyze the sulfur cleanup blank. If surrogate recoveries do not meet the acceptance criteria after reanalysis, the sulfur cleanup blank and all samples associated with that sulfur cleanup blank must be re-extracted and reanalyzed at no additional cost to the Agency.

12.1.3.5.4 If the sulfur cleanup blank failed to meet the criteria used in 12.1.4.4.2, 12.1.4.4.3 and 12.1.4.4.5, then there is an instrument problem. Correct the instrument problem and reanalyze the sulfur cleanup blank.

12.1.3.5.5 If any technical acceptance criteria (Section 12.1.4.4) for sulfur cleanup blank analyses are not met, then the contractor must stop and correct the problem before continuing the analytical sequence. If sample analyses are reported with non-compliant blanks, then the contractor may receive a commensurate reduction in sample price or zero payment depending upon the impact of the non-compliance on data usability.

12.1.4 Instrument Blanks

12.1.4.1 Summary of Instrument Blanks

An instrument blank is a volume of clean solvent spiked with the surrogates and analyzed on each GC column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry over of analytes from standards or highly contaminated samples into other analyses.

12.1.4.2 Frequency of Instrument Blanks

The first analysis in a 12-hour analytical sequence must be an instrument blank. All acceptable sample analyses are to be bracketed by acceptable instrument blanks, as described in Section 10.4.2.l If more than 12 hours have elapsed since the injection of the instrument blank that bracketed a previous 12-hour period, an instrument blank must be analyzed to initiate a new 12-hour sequence.

12.1.4.3 Procedure for Instrument Blanks

12.1.4.3.1 Prepare the instrument blank by spiking the surrogates into hexane for a concentration of 20 ng/ml of tetrachloro-m-xylene and decachlorobiphenyl.

12.1.4.3.2 Analyze the instrument blank according to Section 10.4 at the frequency listed in Section 12.1.5.2

12.1.4.3.3 For comparing the results of the instrument blank analysis to the CRQLs, assume that the material in the instrument resulted from the extraction of a 1 L water sample and calculate the concentration of each analyte using the equation in Section

11.2.1.1. Compare the results to one-half the CRQL values for water samples in Exhibit C (PCBs).

12.1.4.4 Technical Acceptance Criteria for Instrument Blanks

12.1.4.4.1 All instrument blanks must be prepared and analyzed at the frequency described in Section 12.1.5.2 using the procedure in Section 12.1.5.3 on a GC/ECD system meeting the initial calibration and calibration verification technical acceptance criteria.

12.1.4.4.2 The concentration of each of the target analytes (Exhibit C, PCBs) in the instrument blank must be less than 0.5 times the CRQL for that analyte.

12.1.4.4.3 The instrument blank must meet all sample analysis technical acceptance criteria in Sections 11.3.4 through 11.3.7.

12.1.4.4.4 Surrogate recoveries must fall within the recovery limits of 30-150%. In the case of the instrument blank, these limits are not advisory.

12.1.4.5 Corrective Action for Instrument Blanks

12.1.4.5.1 If analytes are detected in the instrument blank at greater than half the CRQL, or the surrogate RTs are outside the RT windows, all data collection must be stopped, and corrective action must be taken. Data for samples which were run between the last acceptable instrument blank and the unacceptable blank are considered suspect. An acceptable instrument blank must be run before additional data are collected. After an acceptable instrument blank is run, all samples which were considered suspect as defined by the criteria described above must be reinjected during a valid analytical sequence at no additional cost to the Agency and must be reported.

12.1.4.5.2 If the surrogate recoveries in the instrument blank do not meet the acceptance criteria listed in section 12.1.4.4.4, first reanalyze the instrument blank. If surrogate recoveries do not meet the acceptance criteria after reanalysis, the instrument blank and all samples associated with that blank must be reanalyzed at no additional cost to the Agency.

12.1.4.5.3 If sample analyses are reported with any non-compliant instrument blanks, then the contractor shall receive a commensurate reduction in sample price or zero payment depending upon the impact of the non-compliance on data usability.

12.2 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

12.2.1 Summary of MS/MSD

In order to evaluate the effects of the sample matrix on the methods used for Aroclor analysis, the Agency has prescribed Aroclor 1254 be spiked into two aliquots of a sample, and analyzed in accordance with the appropriate method.

If the target Aroclor is provided by the RSCC prior to the analysis of samples, then that Aroclor must be used in the matrix spiking solution.

12.2.2 Frequency of MS/MSD Analysis

12.2.2.1 A matrix spike and matrix spike duplicate must be extracted and analyzed with every sample delivery group or with at least every 20 samples per matrix. The Agency may require additional MS/MSD analyses, upon Regional request, for which the Contractor will be paid.

12.2.2.2 As a part of the Agency's QA/QC program, aqueous equipment rinsate blanks (field QC) may accompany soil/sediment/solid samples, water samples and/or oil or oily sludge (waste) samples that are delivered to the laboratory for analysis. The Contractor shall not perform MS/MSD analysis on any of the designated field QC samples.

12.2.2.3 If the EPA Region designates a sample to be used as an MS/MSD, then that sample must be used. If there is insufficient sample volume to perform an MS/MSD, then the Contractor shall contact the RSCC to ascertain an alternate sample to be used for the MS/MSD analysis. The EPA sample numbers, Regional instructions, and date of contact must be included in the SDG Narrative.

12.2.2.4 If there is insufficient sample volume remaining in any of the samples in an SDG to perform an MS/MSD, then the Contractor shall immediately contact the RSCC to inform them of the problem. The Region will approve that no MS/MSD is required, or that the unspiked sample is analyzed at full volume and the MS/MSD is analyzed at reduced volume. The Contractor shall document the Region's decision in the SDG Narrative.

12.2.2.5 The Contractor will not be paid for MS/MSD analyses performed at a greater frequency than required by the contract unless it is requested by the Agency. If the Contractor has a question regarding the frequency, etc., of the MS/MSD analyses for a particular SDG, contact the RSCC for clarification.

12.2.2.6 When a Contractor receives <u>only</u> performance evaluation (PE) samples, no MS/MSD shall be performed within that SDG.

12.2.2.7 When a Contractor receives a PE sample as part of a larger SDG and the Region did not designate an MS/MSD, then a sample other than the PE sample must be chosen for the MS/MSD. If the PE sample is received as an ampulated standard extract, the ampulated PE sample is not considered to be another matrix type.

12.2.3 Procedure for Preparing MS/MSD

12.2.3.1 Water Samples

For water samples, measure out two additional 1 L aliquots of the sample chosen for spiking. Adjust the pH of the samples (if required). Using a syringe or volumetric pipet, fortify each sample with 1.0 ml of the matrix spiking solution (Section 7.2.4.2.1) and 1.0 ml of the surrogate standard spiking solution (Section 7.2.4.1.1). Extract, concentrate, cleanup, and analyze matrix spike and matrix spike duplicate samples according to procedures for water samples in Section 10.1.3.

12.2.3.2 Soil/Sediment/Solid Samples

For soil/sediment/solid samples weigh out two additional 30 g

(record weight to the nearest 0.1 g) aliquots of the sample chosen for spiking. Add 60 g of anhydrous powdered sodium sulfate to each aliquot and mix well. Add 1.0 ml of the matrix spiking solution (Section 7.2.4.2.1) and 1.0 ml of the surrogate standard spiking solution (Section 7.2.4.1.1). Extract, concentrate, cleanup, and analyze matrix spike and matrix spike duplicate samples according procedures for soil/sediment/solid samples in Section 10.1.4.

12.2.3.3 Note: Before any MS/MSD analysis, analyze the original sample, then analyze the MS/MSD at the same concentration as the most concentrated extract for which the original sample results will be reported. For example, if the original sample is to be reported at a 1:1 dilution and a 1:10 dilution, then analyze and report the MS/MSD at a 1:1 dilution only. However, if the original sample is to be reported at a 1:10 dilution and a 1:100 dilution, then the MS/MSD must be analyzed and reported at a 1:10 dilution only. Do not further dilute the MS/MSD samples to get either spiked or non-spiked analytes within calibration range.

12.2.3.4 Oil and Oily Sludge Samples - Waste Dilution

For oil and oily sludge (waste) samples, prepare two additional 1 g aliquots (record weight to the nearest 0.1 g) of the sample chosen for spiking in two precalibrated 20 ml vials. Add 0.5 ml of the matrix spiking solution (Section 7.2.4.2.2) and 0.2 ml of the surrogate standard spiking solution (Section 7.2.4.1.2). Dilute to 10.0 ml with hexane. Extract, concentrate, cleanup and analyze the MS/MSD according to procedures for oil/oily sludge samples in Section 10.1.5.

12.2.4 Calculations for MS/MSD

12.2.4.1 Calculate the concentrations of the matrix spike compounds using the same equations used to calculate target compounds (Section 11.2.1).

12.2.4.2 The percent recoveries and the relative percent difference between the recoveries of the target Aroclor in the matrix spike samples will be calculated and reported by using the following equations:

EQ. 13

$$Matrix\ Spike\ Recovery = \frac{SSR - SR}{SA} \times 100$$

Where,

SSR = Spike sample result
SR = Sample result
SA = Spike added

EQ. 14

$$RPD = \frac{|MSR - MSDR|}{\frac{1}{2}(MSR + MSDR)} \times 100$$

Where,

RPD = Relative percent difference
MSR = Matrix spike recovery
MSDR= Matrix spike duplicate recovery

12.2.4.2 The vertical bars in the formula above indicate the absolute value of the difference, hence RPD is always expressed as a positive value.

12.2.5 Technical Acceptance Criteria for MS/MSD

12.2.5.1 The requirements below apply independently to each GC column and to all instruments used for these analyses. Quantitation must be performed on both GC columns.

12.2.5.2 All MS/MSD must be prepared and analyzed at the frequency described in Section 12.2.2 using the procedure above and in Section 10 on a GC/ECD system meeting the initial calibration, calibration verification, and blank technical acceptance criteria. MS/MSD must be cleaned-up using sulfuric acid and/or sulfur, when required. MS/MSD must be bracketed at 12-hour intervals (or less) by acceptable analyses of instrument blanks and midpoint Aroclor calibration verification standards as described in Section 10.4.2.1.

12.2.5.3 The MS/MSDs must meet all applicable sample technical acceptance criteria as defined in Sections 11.3 through 11.7.

12.2.5.4 The acceptance criteria for MS/MSD recoveries and RPD are given in Table 3. As these limits are only advisory, no further action by the laboratory is required. However, frequent failures to meet the limits for recovery or RPD warrant investigation by the laboratory, and may result in questioning from the Agency.

12.2.5.5 The MS/MSDs must be extracted and analyzed or reextracted and reananlyzed within the contract holding time specified in Section 8.3.

12.2.6 Corrective Action for MS/MSD

Any MS/MSD that does not meet the technical acceptance criteria for MS/MSD must be reanalyzed at no additional cost to the Agency. Both sets of data must be reported.

12.2.6.1 Corrective actions for failure to meet GC/ECD initial calibration and continuing calibration technical acceptance criteria must be completed before the analysis of any QC samples.

12.2.6.2 Corrective actions for failure to meet blank technical acceptance criteria must be completed before the analysis of any QC samples.

12.2.6.3 If the technical acceptance criteria for MS/MSD analysis are not met, the contractor shall determine whether the non-compliance is due to the sample matrix or GC/ECD system problems.

12.2.6.4 If the non-compliance is found to be due to a sample matrix effect, take the following corrective action steps:

- Reanalyze the sample. EXCEPTION: If surrogate recoveries in a sample used for a matrix spike or matrix spike duplicate were outside the recovery limits, then the sample should be reanalyzed only if the surrogate recoveries met the recovery limits in both the matrix spike and matrix spike duplicate analyses.
- If the MS/MSD recoveries/RPD meet the MS/MSD technical acceptance criteria in the reanalyzed sample, then the problem was within the Contractor's control. The Contractor must reanalyze the sample within the contract required holding times. If the reanalysis was performed within holding times, then submit data only from the reanalysis. If the reanalysis was performed outside holding times, then submit both sets of data.
- If the MS/MSD recoveries/RPD fail to meet the acceptance criteria in the reanalysis, then submit data from both analyses. Distinguish between the initial analysis and the reanalysis on all deliverables using the suffixes in Exhibit B.

12.3 SDG-Specific Performance Evaluation (PE) Samples (PEs)

12.3.1 Summary of-SDG Specific PE Samples

The Region I Performance Evaluation (PE) program has two functions, (1) to evaluate laboratory operation and protocols over a period of time, and (2) to provide information on the quality of individual data packages.

12.3.2 Frequency of SDG-Specific PE Samples

12.3.2.1 The Region will submit PE samples with every SDG per parameter and matrix (as available). The Region may obtain these SDG Specific PE samples from either a commercial vendor or from the CLP National Program Office (NPO) which provides PE samples in support of the Superfund program. PE samples provided by the CLP-NPO are referred to as "EPA generated".

12.3.2.2 When the Region submits aqueous trip and/or equipment blanks and/or Performance Evaluation samples (PEs) with soil/sediment/solid or oil/oily sludge samples, then the Contractor shall not perform an MS/MSD analysis on the aqueous matrix (trip blank, equipment blank, PE sample). When the Region submits an aqueous PE sample with aqueous field samples, then the Contractor shall not choose the PE sample for MS/MSD analysis.

12.3.2.3 If the PE sample is received as an ampulated standard extract, the ampulated PE sample is not considered to be another matrix type.

12.3.3 Procedure for Preparing SDG-Specific PE Samples

12.3.3.1 Instructions for preparation of the PE samples will be included with each submission of samples.

12.3.3.2 If PE sample directions do not apply to a PE sample received, then the Contractor must contact the RSCC to ascertain whether or not to analyze the PE sample and/or to obtain appropriate PE sample directions.

12.3.4 Calculations for SDG-Specific PE Samples

12.3.4.1 For EPA generated and commercially prepared PE samples that are submitted with each SDG, the Contractor must correctly identify and quantitate all compounds detected in the PE sample using the criteria presented in Section 11.0 - Data Analysis and Calculations.

12.3.5 Technical Acceptance Criteria for SDG-Specific PE Samples

12.3.5.1 All SDG Specific PE samples must be analyzed under the same GC/ECD conditions set up in Section 9.0 and must meet the same technical acceptance criteria established for sample analysis defined in Section 11.3.

12.3.5.2 EPA-generated PE samples included with the SDG will be evaluated by the Region using a CLP NPO computer program called PeacTOOLs. PeacTOOLs rates the PE sample results based on statistically generated confidence intervals.

12.3.5.3 The results of commercially prepared PE samples will be evaluated using the vendors' statistically generated confidence intervals.

12.3.5.4 Contractor's results on the SDG-Specific PE samples will be evaluated using the most recent Regional PE sample data validation criteria.

12.3.5.5 At a minimum, the PE results will be evaluated for compound identification, quantitation, and sample contamination. Confidence intervals for the quantitation of target compounds are based on reported values using population statistics. The Agency may adjust the scores on any given laboratory evaluation sample to compensate for unanticipated difficulties with a particular sample.

12.3.6 Corrective Action for SDG-Specific PE Samples

12.3.6.1 The corrective actions for PE sample results which do not meet the technical acceptance criteria defined in Section 12.3.5.1 above are the same corrective actions outlined for sample analysis in Section 11.4.

12.3.6.2 If an SDG-Specific PE sample, evaluated as described in Sections 12.3.5.2 through 12.3.5.5 above, indicates unacceptable laboratory performance then the Contractor may be required to reanalyze all samples, standards and QC samples associated with the unacceptable PE sample result (if sufficient volume remains) and/or analyze a new PE sample at no additional cost to the Agency. Unacceptable laboratory performance includes either a TCL false positive result, false negative result, and/or compound misquantitation (reported result exceeds $\pm$ 3 sigma of the spiked compound concentration).

12.3.6.3 SDG-Specific sample results reported with unacceptable PE results shall be subject to a commensurate reduction in sample price or zero payment due to data rejection, depending upon the impact of the non-compliance on data usability.

13.0 METHOD PERFORMANCE

Not Applicable

14.0 POLLUTION PREVENTION

14.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

15.0 WASTE MANAGEMENT

The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

16.0 REFERENCES

16.1 U.S. EPA, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846/8080A, 3rd Edition, September, 1994.

16.2 U.S. EPA, "USEPA Contract Laboratory Program, Statement of Work for Organic Analyses," OLM03.1, Office of Solid Waste and Emergency Response, EPA-540/R-94/073, August, 1994.

16.3 U.S. EPA, "Organochlorine Pesticides and PCBs - Method 608," 40 CFR, Pt. 136, App. A.

16.4 ASTM Annual Book of Standards, "Standard Method for Analysis of Environmental Samples for Polychlorinated Biphenyls," Volume 10.03, Method D 3304.

16.5 U.S. EPA, "The Determination of Polychlorinated Biphenyls in Transformer Fluid and Waste Oils," Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, EPA-600/4-81-045, September, 1982.

17.0 TABLES/DIAGRAMS/FLOWCHARTS

Table 1

Retention Time Windows for
Multicomponent Analytes and Surrogates

Compound	Retention Time Window (minutes)
Aroclor 1016	± 0.07
Aroclor 1221	± 0.07
Aroclor 1232	± 0.07
Aroclor 1242	± 0.07
Aroclor 1248	± 0.07
Aroclor 1254	± 0.07
Aroclor 1260	± 0.07
Aroclor 1262	± 0.07
Aroclor 1268	± 0.07
Tetrachloro-m-xylene	± 0.05
Decachlorobiphenyl	± 0.10

Table 2

Number of Potential Quantitation Peaks

Multicomponent Analyte	No. of Potential Quantitation Peaks
Aroclor 1016/1260	5/5
Aroclor 1221	3
Aroclor 1232	4
Aroclor 1242	5
Aroclor 1248	5
Aroclor 1254	5
Aroclor 1262	5
Aroclor 1268	5

Table 3

Matrix Spike Recovery and Relative Percent Difference Limits

Compound	%Recovery Water	RPD Water	%Recovery Soil/Oil	RPD Soil	%Recovery Oil	RPD Oil
Aroclors	50-150	25	50-150	40	50-150	40